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Report of the Chief of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, 1952

UNITED STATES DEPARTMENT OF AGRICULTURE,
Washington, D. C., September 2, 1952.

Dr. B. T. SHAW,
Agricultural Research Administrator.

DEAR DR. SHAW: I present herewith the report of the Bureau of Agricultural and Industrial Chemistry for the fiscal year ended June 30, 1952.

Sincerely,

G. E. HILBERT,
Chief.

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INTRODUCTION

The Bureau of Agricultural and Industrial Chemistry is a research organization dedicated to the search for new and extended outlets for farm crops. The research program of the Bureau is designed to make the farmer's products more useful or more attractive so that there will be increased demand for them; to process them more efficiently in order to reduce costs and avoid waste; and to make them more stable so that they can be marketed according to demand, thus avoiding seasonal surpluses. As progress is made on this program, the farmer is benefited by receiving higher and more stable prices for his crops, industry and labor are provided with new opportunities, and the consumer can be offered new or improved products, as well as lower prices for existing products.

The research activities of the Bureau are conducted at 4 major regional research laboratories and 11 smaller branch stations. Because of space limitations, it is impossible to describe all the lines of work that have engaged the attention of the Bureau's research personnel during the fiscal year 1952. The items selected for inclusion in this report are grouped under five main headings, four of which indicate the principal outlets in which the agricultural products are utilized; namely, (1) foods and feeds, (2) fibers and fiber products,

(3) medicinals, and (4) industrial products. The fifth group includes basic and pioneering investigations, which play an important role in the efficient utilization of farm crops.

The importance of basic research as a prerequisite to the development of new products for industrial and consumer use cannot be overemphasized. The large chemical companies have been well aware of this for a long time, and many examples could be cited from their experience. One of the most striking is the development of nylon. A large team of research workers spent more than 10 years in basic chemical and physical studies before they were able to synthesize a fiber with properties similar to those of silk and solve the problems encountered in producing this fiber on a commercial scale.

The following examples from the present report indicate the Bureau's realization of the value of basic research.

Practical results of basic and pioneering investigations.—Wheat growers and breeders are receiving valuable assistance in the selection of new varieties of wheat by two new tests—a microscopic test by which the milling quality of the wheat may be predicted after examination of only a few kernels, and a chemical procedure that indicates milling quality on the basis of the acid-soluble pentosan content of the wheat.

Cotton growers and breeders have been given a valuable new method for determining the viability of a given lot of cottonseed (the percentage of seed expected to germinate when planted). This test resulted from an extended study of the composition of cottonseed, which showed that germination decreases as the free fatty acid in the oil increases.

Data on the composition of cottonseed oil are important guides in the selection of oils best suited to the commercial processes used in making shortenings and salad oils. Oils that have the lower percentages of saturated acids are more suitable for processing into winterized salad oils and special shortenings. Bureau scientists have provided a method for estimating the approximate percentages of saturated and unsaturated fatty acids from the iodine values of the oils.

Cotton products and processing procedures for cotton have been improved as the result of new fundamental knowledge of cotton, such as the nature of the primary wall of cotton fiber, the surface-adsorption properties of cotton fibers, and the molecular weight of cotton cellulose.

Apparatus and methods developed for use in radar in World War II have been adapted for investigating the fundamental electrical properties of wool, such as its dielectric constant, a sensitive index of the important interaction between wool and water molecules.

A guide to the preservation of the feeding value of concentrated foods and feeds during storage—particularly at elevated temperatures—has been furnished by fundamental studies on the chemical reactions of their proteins and sugars.

A systematic investigation on the enzymes in certain fruit is supplying data that will aid in explaining the changes which occur during processing and storage, and suggest means for retarding undesirable changes such as softening or rotting and accelerating desirable changes such as ripening and development of flavor.

Information obtained in extended investigations of the proteins of egg white is now being employed in studies on a variety of practical problems. These include (1) causes of the deterioration of shell eggs during storage, (2) effects of changes in egg white constituents on the usefulness of egg products in cookery, (3) effects of pasteurization on egg products, and (4) damage to eggs incurred during dehydration.

Defense research activities.—The Bureau has given priority to research activities that contribute directly to National Defense ever since this policy was enunciated by the President in his letter of July 21, 1950, addressed to all Government agencies. The research program has been changed to place maximum effort on projects of immediate concern to the Departments of the Army, Navy, and Air Force. Substantial progress on some of these projects is recorded in this report.

The urgent need for blood-plasma substitutes or extenders has stimulated research on dextran, one of the most promising extenders now known. Natural dextrans of high molecular weight are produced from glucose by special bacteria. These dextrans have to be partly broken down to a molecular weight that will be suitable for the body's circulatory system. Preparation of dextran for use with blood plasma requires research in bacteriology and biochemistry for the isolation, identification, and culture of dextran-producing bacteria; chemical research on the isolation, purification, and characterization of the dextran fractions; and physical research on the determination of molecular weights, osmotic pressures, and viscosities.

The facilities of the Bureau are ideal for such research, and natural and clinical dextrans have been produced by different strains of bacteria. These dextrans are being evaluated by medical investigators, both civilian and military, selected by the National Research Council. Information on all phases of the manufacture of dextran is being furnished to industrial organizations. All known domestic producers of blood plasma extenders from dextran are now using the micro-organism isolated at the Northern Regional Research Laboratory for the conversion of sugar to natural dextran. Physical methods devised by the Bureau for characterizing the chemical material are particularly effective in accelerating commercial production.

Substantial progress has been made in Bureau laboratories toward satisfying the need of our Armed Forces for concentrated food rations of suitable palatability. The Eastern Regional Research Labortaory is cooperating with the Food and Container Institute of the Quartermaster Corps in making a compressed potato-chip product that contains a good combination of nutrients in concentrated form and remains stable in storage, even at elevated temperatures. The Western Regional Research Laboratory has contributed substantially to the improvement of the commercial production of mashed-potato powder. The Northern and Southern Regional Research Laboratories are developing a global edible spread that can be substituted for butter and margarine and will spread readily at temperatures ranging from 0° to 100° F.

One of the hazards of modern warfare is exposure to fire from many different sources—such as flame throwers, incendiary bombs, atom

bombs, and even flaming gasoline in tanks. Research at the Southern Regional Research Laboratory points the way not only to better protection of combat personnel against fire, but also to better chances for recovery from severe burns.

Added protection against fire is being provided by flame-resistant military clothing. The Southern Laboratory has produced two new flameproofing compounds, which can be applied to cotton fabrics by commercial processing techniques. The excellent flameproofing qualities of the treated textiles are retained even after repeated launderings. A new universal protective dressing for large burned areas on the human body has been adopted by the Office of the Surgeon General for the Armed Forces. Quick and efficient application of this dressing is facilitated by a new body-conforming bandage developed at the Southern Laboratory.

Synthetic lubricants that can be used effectively over wide ranges of temperature are required for both military and civilian uses. The Bureau's Naval Stores Research Division has produced new derivatives from gum turpentine that have the properties needed in turbojet lubricants. These new synthetic lubricants will remain liquid at temperatures as low as –75° F. and are stable at high temperatures.

Conservation of natural resources.—The process developed by the Western Regional Research Laboratory for utilizing pear cannery waste has been demonstrated to be practical and economically sound in commercial operation. The pear pomace and molasses produced by dehydrating the cannery waste are suitable for use in mixed feeds for cattle and sheep and can be readily sold for feed at prices more than sufficient to cover the cost of operation.

A process for separating sugarcane bagasse into pith and fiber, under development at the Northern Regional Research Laboratory, promises to serve a double purpose in the field of conservation. The absorptive capacity of the pith can be used to produce a dry, granular, nonhygroscopic stock feed from blackstrap molasses. The depithed bagasse fiber takes its place with wheat straw as a suitable substitute for wood in the production of pulps for fine papers and newsprint.

Studies at the Eastern Regional Research Laboratory have resulted in a treatment for tobacco stems that renders them safe for use as fertilizer. The stems are highly regarded as a fertilizer and as a conditioner in mixed fertilizers, provided that they are free from infection, such as mosaic virus, which would contaminate the soil. It was found that these stems can be sterilized by heating at 212° F. in the presence of about 32 percent moisture.

Publications and patents.—Further information on the Bureau's work is available in the official publications that appeared during the year. Bureau employees contributed 442 printed and processed publications. Most of them were research papers published in non-Government scientific and technical journals. Usually a limited number of reprints of such papers are bought for distribution to seriously interested individuals and firms as long as the supply lasts.

Information on newly developed processes and products is given in the specifications of the 100 patents granted to Bureau scientists.

More than 25,000 letters requesting information and publications were received by the Bureau during the year.

FOODS AND FEEDS

Fruits

Quality of processed sour cherries affected by many factors

The sour cherry processing industry is becoming increasingly quality-conscious. Occasional lots of cherries are of such poor quality that they are not suitable for processing. Use of new organic fungicides has focused attention on the effect of spray materials on the composition and quality of cherries. It is also realized that practically no factual information is available about the effects of soaking and other preprocessing treatments on the quality and yield of canned cherries.

The Michigan Agricultural Experiment Station, working under an RMA research contract, has just completed a 3-year study of the effect of spray materials on the quality of sour cherries for processing. Wax emulsion sprays when used in combination with fungicides produced abnormally large fruit, with a corresponding reduction in soluble solids and color. Wax sprays have recently been discontinued because of this adverse effect on quality. Copper sprays produced small cherries in dry seasons. With adequate rainfall, there was little if any difference between copper sprays and the carbamate sprays, Fermate and Nabam. It is apparent that the choice of spray material must be governed to some extent by climatic conditions.

The Bureau's Eastern Regional Research Laboratory has made an intensive study of bruising as it affects the quality and yield of canned sour cherries. Most commercially harvested cherries are severely bruised during picking and handling. It is common practice to soak cherries in cold water for 6 to 30 hours prior to pitting and canning. Contrary to the general belief, bruised cherries do not increase in weight during soaking. They increase slightly in firmness, but it was found that this is the result of reduction in temperature. Storage in cold air is just as effective as soaking. Prolonged soaking actually causes serious deterioration in quality, and results in high cullage losses. Cherries may lose as much as 8 percent of their soluble solids and appreciable color when soaked for 24 hours.

Canned sour cherries and many other canned fruits are graded and sold partly on the basis of drained weight. Many lots of processed cherries are too low in drained weight. Recent studies showed that the drained weight may be affected significantly by the extent of bruising, the temperature and length of the period between harvest and processing, and the hardness of the processing water, factors which heretofore have not received much attention. These factors appear to induce changes in the cell walls and in the intercellular cement of the cherry tissues. High drained weight seems to be associated with a high degree of cohesiveness of the tissues. Rather surprisingly, bruising under some conditions increases cohesiveness of the tissues and drained weight. Minimum drained weights are obtained when cherries are processed either immediately after being picked or immediately after being severely bruised. Studies designed to show the interrelationships between the various factors are being continued.

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New kind of apple sherbet has taste appeal

A new use for frozen concentrated apple juice is its incorporation as flavoring in a sherbet. The sherbet, now in its second season of commercial production, was developed at the Western Regional Research Laboratory in research carried out in cooperation with the Washington State Apple Commission to develop new uses for apples not suited for the fresh market because of poor color or shape. A large dairy company test-marketed the product in California in 1951, and again produced and marketed it in 1952, both as a sherbet and in an ice-cream-and-sherbet pie.

The new product is based on studies at the New York Agricultural Experiment Station, where concentrated apple juice was tested for use in ice cream. The sherbet contains small chunks of fresh apple impregnated with a fourfold concentrated apple juice, which gives it additional eye and taste appeal. It has a definite apple flavor, and the apple chunks provide a pleasing texture. Because the apple chunks impregnated with concentrate have a soluble solids content of more than 26 percent, they are not made icy by the low temperature.

Preparation of the full-flavored concentrate has been described in Bureau publications. Briefly, it consists in removing the volatile flavoring constituents from the juice, separately concentrating the two portions to one hundredfold and fourfold, respectively, and recombining them. The mixture for flavoring the sherbet is made by combining 1 pound of concentrate with 1 pound of diced fresh apple, soaking the submerged pieces for 20 minutes, and then freezing. To delay enzymatic darkening of the apples, 0.2 percent of ascorbic acid is added to the concentrate. For use, the concentrate is drained from the thawed mixture and added to the sherbet mix before freezing. The apple chunks are added from the standard fruit feeder. A satisfactory sherbet contains 12.5 pounds of drained concentrate and 13 pounds of drained apple chunks per 100 pounds of finished sherbet.

Since a concentrate of apple juice is used, the amount of fruit utilized per unit volume of sherbet is much larger than with usual fruit flavors. For a sherbet of 60 percent overrun, approximately 500 pounds of fresh apples are required, for the concentrate and apple chunks, for 100 gallons of finished sherbet. In spite of the large amount of fruit used, the cost of flavoring ingredients appears to be about equal to that for other sherbets, or slightly less.

Better apple juice now possible from Delicious variety

The quality of apples used for juice is important. Information is limited, however, on the effects of factors such as maturity, grade, storage, and preprocessing treatment of apples on the quality of juice made from them. An investigation of this problem is in progress at the Fruit and Vegetable Products Laboratory in Prosser, Wash., in cooperation with the Washington State Tree Fruit Experiment Station at Wenatchee. Standard Delicious apples have been investigated in the first phase of this study because of the importance of this variety in the Pacific Northwest.

Ripening after harvest was found to be the most important factor in improving the juice of the Delicious variety. Fruit pressed soon after harvest yielded juice of poor quality. To obtain juice having a characteristic Delicious flavor, at least 2 weeks of ripening in common

storage was required. This was true even for fruit allowed to ripen on the tree for 16 days after normal harvest. The flavor continued to improve as the ripening period was increased to 4 weeks. Improvement in flavor was accompanied by a marked increase in sugars and loss of starch. The increase in sugars ranged from about 2 to 3 percent, depending on the ripeness of the fruit when harvested.

The maturity of the fruit at harvest is another important factor in the quality of the juice. Juice with the poorest flavor was obtained from fruit harvested at early-season commercial maturity. Juice of good flavor could be produced from such fruit, however, by proper ripening after harvest. The best-flavored juices were obtained from fruit that had remained on the tree for 16 days after the normal harvest period and then ripened for 2 to 4 weeks. This finding indicates the desirability of first harvesting only the well-colored fruits and leaving the poorly colored fruit on the trees for later harvest as juice fruit. In fact, this practice has been considered by various growers as a means of avoiding heavy packing-house handling costs for this lower grade fruit.

The juice quality of Delicious apples was maintained for periods up to 4 months by holding the fruit in commercial cold storage, and juices of good quality were obtained from apples held in cold storage as long as 6 months. Although the fruit ripened considerably during cold storage, juices of improved flavor were obtained by allowing it to ripen for 2 weeks after removal from storage. Juices obtained from Fancy to Extra Fancy grade fruit were slightly higher in sugars and had a slightly better flavor than those prepared from C grade fruit of the same harvest and with similar handling. These differences were not so pronounced as those resulting from postharvest ripening, and good juices were obtained from C grade apples picked during or after the normal harvest period and then properly ripened.

Orange juice powder with good shelf life produced

At the Western Regional Research Laboratory an orange juice powder has been prepared that has sufficient storage stability to meet the requirements for domestic or military use, even at high temperatures. When mixed with water, it is readily reconstituted into a palatable beverage.

The process for the new product uses concentrated orange juice similar to that used for commercially packed frozen concentrate. Low-conversion corn sirup solids are added to the concentrate to reduce the hygroscopicity of the product. This mixture is then vacuum-dried at moderately low pressures; the more expensive high-vacuum or "freeze drying" process is unnecessary. Vacuum-drying is conducted in such a way that the concentrate expands or "puffs" during drying. The resulting open structure makes it possible to dry the material rapidly and at a temperature low enough not to cause significant changes in flavor. The powder, prepared by crushing the dried material to suitable size, retains the porous open structure, which permits rapid reconstitution with water. Since reconstitution is much more difficult when the powder is too fine, attaining the appropriate particle size is an important part of the process.

The original orange flavor, most of which is lost during dehydration, is replaced by adding a small quantity of specially prepared

natural orange oil to the powder. The orange oil is first incorporated in an edible solid carrier to prevent contact of the oil with oxygen; this minimizes changes in flavor of the oil during storage.

The orange powder has a storage life at 100° F. of more than 6 months, with little change in vitamin C content and only minor loss of carotene (provitamin A). Since storage studies have not been completed, definite information on storage life at 70° is not available. Deterioration at this temperature, however, is extremely slow. This excellent stability is achieved in large measure by a packet of desiccant placed in each can of powder. The desiccant removes substantially all remaining moisture from the powder after it has been packaged.

Flavor of frozen orange juice concentrate fortified by orange oil

During 1951 consumers spent close to $200 million for frozen concentrated orange juice. Commercial use of this revolutionary method of processing oranges has developed steadily since 1946. In 1947–48, 2.4 million gallons was produced; in 1948–49, 12 million gallons; in 1949–50, 25 million gallons; in 1950–51, 35 million gallons; and in 1951–52, 48.5 million gallons (estimated).

Because manufacture of frozen orange juice concentrate is now a well-established large industry, even small improvements in methods are significant. In making the frozen concentrate, volatile flavoring constituents are lost during the evaporation. To restore the flavor, fresh single-strength orange juice is added to the concentrated juice, diluting it from about 55° to 42°–43° Brix. The overconcentrating required because of the addition of fresh juice is expensive. Moreover, addition of the single-strength juice does not return all the flavor lost during concentration.

Studies at the Fruit and Vegetable Chemistry Laboratory in Pasadena, Calif., showed that the flavoring materials lost during evaporation are mainly volatile oils and that more than 90 percent of these oils have their origin in the peel. Guided by this basic knowledge, the investigators have developed a method for fortifying the flavor of orange concentrates by adding small amounts of good-quality orange oil in place of the large amounts of single-strength cut-back juice. With this method it is unnecessary to overconcentrate; evaporation is stopped at 43° Brix. If desired, however, evaporation could be carried to 58° to 59° Brix, and the resulting concentrate, flavor-fortified with orange oil, could be marketed as an approximately 6:1 concentrate instead of the present 4:1 product. This further concentration would mean considerable savings in containers and refrigerated storage space, as well as in the cost of shipping. Further work is under way on commercial application of these findings and on chemical variations in orange oils that determine their suitability for fortifying flavor.

Another obvious source of orange flavor is the mixture of oils that volatilize from the juice during concentration. If these oils could be successfully and economically trapped, processors would be able to return to the final concentrate the same flavoring oils present in the original juice. A refrigerated brine absorber has been developed to trap the volatile oils lost during evaporation. The flavor of concentrates can be fortified also with orange puree, which contains approxi-

mately 0.5 to 0.75 percent oil, in place of the single-strength cut-back juice. Research is under way on both trapped volatile oils and orange purees as flavors for concentrates. Tests showed that concentrates fortified with orange oils and purees are stable at temperatures used in commercial storage.

New equipment developed for flash-heating and concentrating juices

Work was continued on the development of new equipment for the rapid heat-processing of fluid foods. Two units, a direct steam-injection heater and a combination evaporator (a single-pass, tubular evaporator incorporating the steam-injection heater as a preheater), were described in the 1950 report. These equipment units have been used successfully for the heat-processing (pasteurizing, essence-stripping, concentrating, deaerating, and enzyme-inactivating) of fruit juices (grape, tomato, and six varieties or types of apple), fruit purees (apricot, peach, pear, and berry), pea puree, and milk. The equipment is highly effective; many heat-sensitive products can be processed without detectable impairment of flavor. The system is largely self cleaning, showing little tendency toward fouling of heat-transfer surfaces.

In view of the highly successful results obtained with the original model of the single-pass concentrator, a new model was constructed that has more than eight times the heating surface of the original. Total processing times characteristic of the original model were retained, that is, less than 1 second is required to heat, concentrate, and cool the material in process. The new concentrator has been tested in the pilot plant for producing apple juice and grape juice. Evaporation rates per unit of surface area in the concentrator are as much as 250 percent greater than in the previous model. Evaporation rates per square foot of heating surface range from 3 to 10 times the rates in conventional evaporators.

There is active commercial interest in this work, and the new equipment is now in operation in several commercial plants. Single-pass evaporators based on this unit have been applied successfully on a commercial scale for processing tomato juice, apple juice, and pear puree. Two plants are using steam-injection heating to control cloud stability in frozen concentrated orange juice. Other evaporators will be in operation during the current season. The processing capacity of present installations ranges from 500 to 6,700 gallons of feed an hour, with evaporation rates of 800 to 28,000 pounds of water an hour. The concentrators can be operated independently, or they can be used in combination with standard vacuum pans to reduce steam consumption. The new equipment is suitable for either type of installation; the choice depends on local economic conditions.

Florida citrus industry revitalized by frozen concentrates

The research team that developed the basic process for producing frozen concentrated orange juice, now in wide commercial use, was given the Department of Agriculture's Distinguished Service Award, the Department's highest honor, in May 1952. Designated the Florida Citrus Concentrate Group, the team was composed of three scientists from the Bureau and four from the Florida Citrus Commission.

The research conducted by this group resulted in an outlet for large tonnages of oranges and a nutritious frozen product widely used in the home and in restaurants.

Instead of the usual 10 to 15 years from laboratory success to commercial production, development of the product was condensed into a 5-year period. Production of frozen concentrated orange juice in Florida increased from a meager 226,000 gallons in 1945–46 to 21,647,000 gallons in 1949–50, and in 1951–52 to about 44,000,000 gallons. By 1955, Florida's orange-bearing acreage alone is expected to be about 500,000 acres. The popularity of frozen orange juice concentrate is undoubtedly one of the major factors contributing to the present over-all expansion of the citrus industry in Florida. There is good reason to believe that the production of frozen orange concentrate will continue to increase as processing refinements yield better quality, as new markets are found, and as new methods permit storage by the consumer at higher temperatures. Application of the process to other fruit juices promises to provide still greater markets for grapefruit, apples, and other fruits.

During the last 10 years, Bureau research has contributed considerably to the improvement of citrus-processing methods and to the establishment of proper conditions for storing and handling the concentrate. The most important contributions were made in the U. S. Citrus Products Laboratory, Winter Haven, Fla., by the cooperative efforts of Bureau and Florida Citrus Commission scientists. Methods of concentrating the juice were investigated, and a method for preparing full-flavored fruit juice concentrates was developed. A public-service patent has been issued on this process.

Stabilization of citrus concentrates investigated for storage above 0° F.

During the last 5 or 6 years, the citrus industry has made phenomenal progress in development of frozen concentrated citrus juices. Florida orange production alone reached 78,900,000 boxes in 1951–52, approximately two-fifths of which were used in the processing of 44,000,000 gallons of frozen concentrated juice.

Since frozen concentrated orange juice is not pasteurized, it must be kept at freezing temperatures to maintain fresh-fruit flavor during transportation and storage. A temperature of about 0° F. is needed to keep deterioration at a minimum, and since this condition of storage is not always available, it is highly desirable to develop concentrates that will be stable at higher temperatures. As a step toward this objective, studies were made at the Bureau's Citrus Products Laboratory, Winter Haven, Fla., to determine the effects of various temperatures on orange juice at several levels of concentration—single strength and twofold, fourfold, and sixfold. Evaluation of the treatments was based on pectinesterase (enzyme) activity, cloud stability, and bacteriological plate counts.

There was a regular decrease in pectinesterase enzyme activity with increase in treating temperature in the range of 120° to 160° F. Heating at 160° inactivated 89 to 95 percent of the pectinesterase originally present. Increasing the temperature from 160° to 180°, resulted in little change in the residual activity, 94–96 percent of the

enzyme being inactivated. Heating at 190° inactivated 97 percent or more; 200° caused not less than 98 percent inactivation. There appeared to be little advantage in heating to 180° instead of 160°.

Plate counts of micro-organisms on Lindegrin's agar indicated that numbers decreased with increasing temperatures up to 150° F. With treatments to 160° and above, there were further reductions, but the differences were not significant.

Clarification—or loss of "cloud"—seriously decreases the attractiveness of orange juice. This undesirable process, which sometimes occurs in juices reconstituted from concentrates, can be retarded by suitable heat treatment. The loss or degradation of the cloud, measured at intervals during storage at 35° F., indicated that stability was not attained by heating at 140° or less. Heating at 160° to 180° stabilized the cloud in sixfold concentrate products only, whereas heating to 190° and 200° stabilized the cloud in all products, including single-strength juice, and twofold and fourfold concentrates.

Although the rate of enzyme inactivation appeared to be similar for any given temperature regardless of concentration, stability of the cloud was attained in sixfold concentrates at lower processing temperatures than were required for less concentrated juice.

Studies are being continued to develop a product that does not require 0° F. for effective preservation during storage.

Remedy sought for off-flavor and odor in canned citrus juices

In spite of the recent rapid expansion in production of frozen concentrated orange juice, canned single-strength juice is still commercially important. It provides consumers with the principal food and vitamin values of citrus fruit without serious loss, but it does not have the excellent flavor of frozen concentrate. Furthermore, the general practice of shipping and storing single-strength canned juice at room temperature aggravates the problem of off-flavors, because the flavor of most, if not all, citrus juice deteriorates in a comparatively short time when the juice is stored at room temperatures.

At the Bureau's Citrus Products Laboratory at Winter Haven, Fla., progress was made in discovering the cause of changes in the constituents of citrus juice during the development of off-flavor and odor. To determine what causes juice to darken, lose ascorbic acid (vitamin C), and develop a sharp, pungent disagreeable flavor, the investigators added different potential precursors—substances from which off-flavors or odors might develop—to whole juice, filtered juice, and synthetic juice. These additives included juice lipid (an oil fraction), a phosphatide, whole-peel oil, terpeneless peel oil, limonene, carotene, limonene plus carotene, and constituents of the lipid such as unsaponifiable matter, fatty acids, choline, ethanolamine, and volatile lipid material. The juices were examined at regular intervals during storage.

These studies indicated that the limonene of the peel oil plays an important role in the development of the characteristic sharp flavor but that some other factor, possibly the dispersing effect of the suspended matter or the lipid phosphatides, is also involved, because whole juice deteriorated much more rapidly than filtered or synthetic juice that contained the same amount of peel oil. Peel oil, from which the terpene constituent had been removed by adsorption, caused off-

flavors in synthetic juice, but they were not the characteristic off-flavors that develop in whole juice.

The problem is important, and it will be studied further in an effort to develop single-strength juice of improved keeping quality.

New frozen limeade base shows promise

A delicious new frozen limeade base developed by the United States Citrus Products Laboratory at Winter Haven, Fla., has advantages over the present commercial product. Since it is more highly concentrated, it is less costly to ship and store. The flavor quality of the product can be standardized easily by adjusting the sugar-acid ratio and the peel-oil content. By using their present equipment, manufacturers can prepare this limeade as a sweetened eightfold concentrate or as an unsweetened sixteenfold concentrate.

A limeade concentrate, prepared by adding sugar to fresh whole lime juice, is available on the market. This can be made into an acceptable beverage by adding three volumes of water. The new sweetened limeade base developed at the Citrus Products Laboratory is prepared by concentrating the juice of fresh limes and then adding sugar and fresh unpasteurized juice to restore the fresh-fruit flavor. The cut-back technique of adding fresh juice to concentrated juice is the basic process employed so successfully in the production of frozen orange concentrate.

Eightfold limeade base is prepared by evaporating fresh lime juice under vacuum to slightly less than one-half the original volume, then adding the proper amount of sugar and fresh juice. Little, if any, vitamin C is lost in processing; the concentrated product usually contains about 24 milligrams of ascorbic acid per 100 milliliters. A delicious limeade beverage is obtained by mixing the concentrated base with seven parts of water. Optimum flavor results when the sugar density is about 10.5° Brix, and the citric acid content about 0.7 percent, giving a sugar-acid ratio of 14 to 1.

A sixteenfold limeade base is also easy to pack. It consists of highly concentrated juice and fresh unpasteurized juice, to which a little sugar is added. Since this type of product occupies only half the volume of the eightfold base, it has an advantage where refrigerated storage space is at a premium.

In the 1950–51 season, Florida produced 280,000 boxes of limes, of which 74,000, or 26 percent, were processed. Only a small percentage went into frozen concentrate. The commercial success of frozen orange concentrate and of frozen lemonade and limeade products clearly indicates the commercial possibilities of the compact eightfold and sixteenfold frozen limeade bases for both civilian and military uses.

Vegetables

Potato chips compressed into bars for military use

Potato chips are popular with the age group predominant in the Armed Forces. This food product has not been used as a military ration, however, because of its excessive space requirements. It was found at the Bureau's Eastern Regional Research Laboratory that potato chips can be crumbled and pressed into bars that occupy about one-twentieth of the original volume. Pressures of about 4,000

pounds p. s. i. g. produced the desired binding without excessive loss of oil. Binding can be further improved by using high-melting hydrogenated vegetable oils in the frying process.

In addition to taste appeal, the potato-chip bars have a good combination of nutrients in concentrated form. Essentially all the nitrogen compounds, carbohydrates, and mineral constituents of potatoes are retained. Since the bars contain about 60 percent potato solids, 38 percent fat, and only 2 percent moisture, they possess the high caloric density required for military rations.

In storage stability tests, conducted in cooperation with the Food and Container Institute of the Quartermaster Corps, potato-chip bars (3 by 1 by 5/8 inches) containing a high-melting hydrogenated vegetable oil and having low moisture contents were resistant to deterioration for 6 months at 100° F. To provide a product having the nutrients and stability required for a survival ration bar, the Laboratory is now investigating the possibility of incorporating protein supplements. Civilian acceptance of this product is also being investigated.

Tests for maturity in frozen peas evaluated

One of the most important factors in the quality of frozen peas is maturity. Good peas must be tender and succulent; tough starchy peas are not desirable. Since this factor is part of the eating quality, it can be measured directly only by human tasters, but reliable appraisals by panels of judges are expensive, time consuming, and in some cases impractical. Objective tests of maturity are needed not only for the control of product quality in the plant and for grading purposes but also for research leading toward improvement of processing and storage.

Many objective tests for maturity of frozen peas have been proposed, but not all have been compared with each other or with the fundamental criterion—appraisal by a taste panel. Indeed, a convincing study could not have been made until a few years ago because methods of panel appraisal available were unreliable, as compared with those in use now.

The Utah Agricultural Experiment Station, under a research contract, and the Western Regional Research Laboratory have undertaken a cooperative study to evaluate the most promising of the tests. In each of the three seasons 1949–51, the Utah Station grew, harvested, and shipped by air to the Western Laboratory pod peas of two freezing varieties, each harvested on four different dates. The Thomas Laxton variety was grown all three seasons; the Rondo, Dark Green Perfection, and Oracle varieties were each grown one season. The Station also made various physical and chemical determinations on the raw peas. When they arrived at the Western Laboratory, the peas were promptly shelled, divided into groups according to size, processed, and frozen under carefully controlled conditions.

About 68 samples, representing 4 varieties, 3 seasons, and various harvest dates and size groups, were used. Subsequently these samples were appraised by a taste panel under conditions that assured a high degree of precision. In addition, six objective tests were made; a seventh was abandoned after the first season because it proved unreliable.

The results clearly indicated the superiority of the alcohol-insoluble

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solids determination as a measure of maturity, as judged by tasters. Although slow, this test can be recommended for research purposes, and it might serve as the basis for a rapid test suitable for control and grading. One mechanical test (tenderometer value) on the cooked peas also showed enough promise to merit further work.

The test currently used for control and grading purposes consists in determining the percentage of peas, with skins removed, that sink in salt solutions of specified concentrations within 10 seconds. A modification of this test was developed in which the substitution of sugar solutions for brines makes the time factor less critical. A vacuum treatment elminates the tedious operation of removing the skins and makes it convenient to test a larger and more representative sample.

Quality of dry beans during storage affected by temperature and moisture

Dry beans, one of our more stable foods, slowly deteriorate during storage, but the slowness of the changes and the lack of simple methods for measuring loss of quality have been limiting factors in detecting and evaluating this deterioration.

The effect of the relative humidity of the storage atmosphere on the equilibrium moisture contents of many grains has been reported. To establish the relationship for beans, studies were carried out at the Western Regional Research Laboratory on six varieties, most of which were furnished by the Idaho Agricultural Experiment Station. All varieties behaved similarly. Continuous storage above 75 percent relative humidity at 77° F. resulted in moisture contents of about 20 percent, sufficient to support growth of molds.

The stability of many "dry" foods during storage is greatly affected by their moisture contents, and beans were found to be no exception. California small white beans, adjusted to five moisture levels within the commercial range of 9 to 17 percent, were stored at 77°. Five months of storage produced significant changes in both the flavor and the texture of the sample with the highest moisture content, as judged by a trained taste panel. After 1 year of storage, the sample with 17 percent moisture content was completely unacceptable, and the samples with 13 to 15 percent moisture had deteriorated significantly in flavor and texture. Changes in composition occurred simultaneously. There was a large increase in the free acidity of the fat fraction of the beans and a loss in phosphatase and catalase. These sensitive enzymes were used as indicators of physiological change. Such changes might furnish objective means for estimating the quality of stored dry beans.

Red Mexican, Great Northern, and Pinto varieties, containing 9 to 10 percent moisture, were used in studies of the effect of storage temperature on retention of quality. Samples stored at 77° and 100° F. were compared with controls stores at −30°. After 2 years' storage at 77°, only slight changes had occurred, but at 100° samples had deteriorated markedly in both flavor and texture. This change was also accompanied by an increase in the free acidity of the fat fraction and a decrease in enzyme activity.

The atmosphere had little effect on the quality of beans stored for 2 years. Red Mexican and Great Northern beans showed about the

same degree of deterioration whether stored in air, oxygen, nitrogen, carbon dioxide, or vacuum. Oxygen did not accelerate loss of quality.

The results of these studies indicated that for maximum stability of dry beans during storage, the relative humidity should not exceed 60 percent (corresponding to 13 percent moisture content), and the temperature should not be above 77° F. At 9 to 10 percent moisture contents, packing in an inert atmosphere does not increase stability.

Mashed-potato powder made by improved procedures

Research initiated at the Eastern and Western Regional Research Laboratories after the outbreak of hostilities in Korea in 1950 has led to improved methods for making dried mashed-potato powder (potato granules). An improved drier was designed, the effects of several process variables on the quality of the product were determined, and an objective method was developed for evaluating the product. In addition, a novel process for removal of water from food products has been applied on a laboratory scale in preparing potato granules.

To reduce mechanical and heat damage in the manufacture of potato granules, an airlift drier having some unusual features was designed. The product is dried while being carried upward in a vertical column by a stream of hot air. The column is expanded at the top to reduce the velocity of the air. Thus large particles remain in the drier longer than small particles, and adjustment can be made to prevent overheating of small particles while large particles are dried to a relatively low moisture content. One of the improved features is a collector that provides for a single change of direction for the fragile granules while they are carried at a relatively low velocity. The product is thus subjected to a minimum of impact and abrasion damage. Several processing plants have incorporated this airlift drier in production lines for conveying and cooling as well as drying. One firm has a pilot-plant drier for developmental studies.

In a study of process variables, granules were produced by drying (in two stages) the moist mixture obtained by adding previously dried and size-graded granules to freshly cooked mashed potatoes (the "add back" process). After the first stage of drying, the product was separated into three fractions by screening. The small proportion of relatively coarse particles was rejected. The intermediate-size particles, together with a portion of the finest particles, were used as the "add back" material. The remainder of the fine fraction, the product to be packed, was brought to the desired moisture content by a second stage of drying. Optimum distribution of intermediate and fine fractions was found necessary to keep the system operating satisfactorily.

A critical factor is the moisture content of the moist granular mixture before drying. This can be adjusted either by changing the "add back" ratio or by changing the moisture content of the "add back" fraction itself by varying the severity of the first stage of drying. Within the range of moisture contents investigated (35 to 45 percent), better distribution of particle size was obtained at the lower moisture levels. Distribution was also best when the mixture was "tempered" for several hours before being dried.

Two important qualities of the final product are also affected by the moisture content of the mixture of fresh mashed potatoes and

"add back" material. The lower this moisture content, the less will be the amount of extractable starch in the product. This is important because a high content of extractable starch causes an undesirable pasty texture of mashed potatoes prepared from the product. In addition, the lower the moisture content of the mixture, the higher will be the packing density of the dry product. The density of the product is an important factor for military subsistence uses, in the saving of space and tin cans. Higher packing density is also associated with a lower drying temperature in the first stage of drying, within the air temperature range of 212° to 392° F. Application of these findings to commercial operations has essentially overcome difficulties in attaining a uniformly high net weight of product in the standard package.

Mechanical action during the production of potato granules may cause rupture of the cell walls. On rehydration, starch is released from these damaged granules, causing undesirable pastiness. Since the release of starch indicates damaging action in the manufacturing operations, a method was developed for measuring the starch easily extracted from potato granules by hot water (150° F.). This method is particularly useful in evaluating processing equipment for damaging action. It has been adopted by processors, and in two processing plants the data obtained led to the installation of new systems that reduced mechanical damage to the product.

In the new process developed on a laboratory scale for the preparation of potato granules, cooked mashed potatoes are dehydrated by batchwise or continuous extraction with water-soluble organic solvents such as acetone, methyl alcohol, and ethyl alcohol. The potato granules, obtained after removal of the solvent-water liquid phase by filtration or centrifugation, are finally dried by mild heat treatment.

The solvent-dehydrated product differs from the granules obtained by the current commercial heat-dehydration process in that it is lighter in color, blander in flavor, and more resistant to deterioration at room temperature. It also has a lower bulk density than the product of the "add back" process. By the addition of water at any temperature, it can be reconstituted to a product that closely approximates freshly mashed potatoes in consistency.

Pectic enzyme studies lead to improved methods for production of tomato paste

Tomato paste is sold commercially for use in manufacturing soups, catsups, and sauces, and for home uses. With the exception of paste sold for remanufacture into soups, one of the most important factors of quality determining the market value of a paste is consistency. For many years, processors have had difficulty in controlling the consistency of tomato paste during manufacture. It has long been the practice in industry to attempt to increase the consistency of thin pastes by evaporating them to a solids content of more than 25 percent. (A solids content of not less than 25 percent is legally required.) As a result, yields of tomato paste per ton of fruit are reduced, and usually little increase in consistency is realized.

The chemistry and technology of tomato paste production are being studied at the Fruit and Vegetable Chemistry Laboratory in Pasadena, Calif., with particular reference to the factors that affect

consistency. Because peptic substances contribute to consistency, methods were developed for measuring and characterizing them. These methods were designed not only to determine the amounts of these substances present but to distinguish between the different types of pectic substances that might occur naturally in the tomato or might result from enzymatic breakdown.

In cooperation with commercial processors, studies were made of the production of tomato paste in about 10 plants in southern and northern California. Samples of whole tomatoes, crushed tomato macerate, and tomato paste were taken at various stages of processing. The temperatures of the samples were recorded at the time of sampling, with particular attention to the preheating temperature employed after the fruit was crushed. It seemed reasonable to suspect that an inadequate preheating temperature would result in failure to inactivate pectic enzymes in the tomato, with resulting destruction of pectic substances during processing. This loss of pectic substances might contribute to low consistency in the tomato pastes.

It was found that pectic enzymes in tomato macerate and paste were not completely inactivated during processing unless a preheating temperature of 185° F. was attained. Pastes prepared with preheating temperatures below this value lacked pectic substances and were low in consistency. Pastes prepared from tomato macerates rapidly preheated to 185° or higher had a relatively high content of pectic substances and high consistencies.

Studies on the distribution of pectic enzymes in the whole tomato showed that a large proportion of these enzymes is localized near the surface of the fruit (pericarp). When whole tomatoes were treated with steam or hot water immediately before the regular preheating ("hot break") process, the enzymes were inactivated more rapidly and more effectively. This treatment resulted in greater retention of pectic substances and produced pastes of higher consistency than were obtained when only "hot break" heating was used.

These studies demonstrated that retention of pectic substances during processing is an important factor contributing to the ultimate consistency of tomato paste. It is now possible to define more accurately the optimum processing conditions for inactivating pectic enzymes and to furnish the industry with the necessary information for improving their processing methods.

Sugar and Sirups

Ultrarapid evaporator makes fancy maple sirup from ordinary sap

One of the principal problems of the maple sirup industry is the lack of control of the color and flavor of the finished sirup. Maple sirup of standard density and without off-flavor is graded on the basis of color, the lighter grades commanding the higher price. Sap collected late in the season usually darkens so much during evaporation in the customary open fuel-fired pan that the sirup can be sold only at a reduced price. A method of reducing and controlling the darkening incident to evaporation is therefore desirable.

The single-pass, tubular, steam-heated rapid evaporator developed at the Eastern Regional Research Laboratory for recovering essences

from fruit juices has been adapted for this purpose. Because of the short heating period required, it concentrates maple sap to sirup with little of the usual darkening. A chamber of adjustable volume then holds the sirup at or near its boiling point for whatever time may be necessary to develop color and flavor. The apparatus can thus be controlled to produce sirup of uniform light color from saps having different characteristics.

The turbopreheater, evaporating tube, and separator are of the same design as those used in the essence-recovery apparatus. Since the preheater and evaporator are steam-jacketed, a steam boiler is required. A pump is also needed to feed the sap to the apparatus at a constant rate.

It is not expected that the average farm producer of maple sirup will find it feasible to install such equipment. The method is intended for use at a central cooperative or commercial evaporating plant. To reduce hauling costs and sterilize the sap, farmers could partly evaporate the sap in their open pans, reducing it to a fraction of its original volume.

Work is now in progress to develop the process and equipment to the stage where definite recommendations can be made.

New products made from high-flavored maple sirup

The process for making high-flavored maple sirup, described in the 1951 report, for which public service patent U. S. No. 2,549,877 was issued, is now being used commercially to produce several carloads of high-flavored maple sirup annually. In addition to its use in making blended cane sugar sirups, it is the basis of two new food products.

Corn Sirup-Maple Sirup Blend.—At the annual meeting of the National Food Brokers Association in January 1952, a new table sirup was introduced that consists of 50 percent corn sirup, 37½ percent cane-sugar sirup, and 12½ percent high-flavored maple sirup. This is the first utilization of corn sirup as a base for a maple-blended table sirup. It is estimated that during the first year (1952) production will exceed 34,000 cases of twenty-four 12-ounce bottles.

The Bureau's contribution has been fully acknowledged in the commercial prospectus describing this new sirup.

Maple Sugar Concentrate.—The other new food product developed from high-flavored maple sirup is a 3-ounce maple cake (sugar) concentrate. It was introduced to the food trade in February 1952 at Minneapolis, Minn. This concentrate, made by reducing high-flavored maple sirup to solid form, is easily blended with cane sugar and water to produce a table sirup almost identical with pure maple sirup in both flavor and color. The high-flavored maple sirup cake, which eliminates keeping and storage problems, has proved to be a popular food item. Because of its compact form and keeping properties, it is of interest to the Armed Forces.

New dial maple sirup thermometer proves useful to farmer-producers

A new maple sirup thermometer was announced in the 1951 report. This reset, dial-type thermometer, by which compensation can be made for barometric effects on boiling temperatures, provides the farmer-producer with the means for making maple sirup of exactly standard density. It has now been given extensive field tests by maple

sirup producers. In each test, it won high acclaim for its (1) readable open scale, (2) high precision and sensitivity, (3) easy adjustment for barometric compensation of boiling point, (4) ease of attachment, and (5) unrestricted reading. Mounted outside the evaporator, it is away from the steam. Because of the successful field trials, it will be included as a stock catalog item by the largest manufacturer of maple sirup equipment and will be offered as optional equipment on all their new evaporators.

Lactic acid in processing liquors indicates loss of beet sugar

During the past 3 years, a basic study has been under way at the Western Regional Research Laboratory on the composition of sugar beets and their processing liquors. Further knowledge of the compounds in beet juice will provide a sounder basis for work directed toward improving the technology of the beet-sugar industry.

Investigations of this type sometimes yield unexpected dividends. It was found that the processing liquors contain lactic acid to the extent of 0.1 to 1.0 percent of the total solids, although it is not usually considered a normal constituent of plant tissue. Tests indicated that beet tissue contains much smaller quantities than appear in the processing liquors. This finding was further confirmed recently by analysis of freshly sliced cossettes that had been immediately sterilized in boiling 70 percent alcohol. At the same time, samples of diffusion liquor were collected from four factories in the principal beet-producing areas and immediately concentrated *in vacuo*. The lactic acid amounted to less than 0.01 percent of the total solids in cossette juice but 0.17 to 0.4 percent of those in diffusion juice.

To determine whether lactic acid is developed during sampling and evaporating, samples of thick juice and molasses were collected from one factory. These liquors were sterile at the time of collection and were preserved with antiseptic. The lactic acid was 0.2 percent of the total solids in the thick juice and about 2.0 percent of the total solids in the molasses, indicating that lactic acid is produced by microbial action during factory operations.

In the past, fermentation during diffusion has been indicated by a decrease in the pH value, but because of the high buffering capacity of beet juice, about 0.2 percent of lactic acid is necessary to decrease the pH by one-half unit, and consequently, antiseptics have been added after significant fermentation has occurred.

The finding that lactic acid is produced during factory operations is significant to the beet industry, because even 0.1 percent of lactic acid produced by fermentation means a loss of about 0.1 percent sugar. In addition each pound of lactic acid causes at least a pound of sugar to become molasses because of its interference with sugar crystallization. In a factory that slices 2,000 tons of beets a day, there would be a daily loss of 1,200 pounds of sugar. For the entire material production, this loss would amout to 2,000 tons of sugar a year, which, when adjusted for the price of molasses, would be valued at about 300,000 dollars.

The need for a rapid method for determining lactic acid is apparent. Such work is under way, and members of the sugar industry are kept informed of progress by reports and conferences. Prevention of loss of sugar by economical, adequate control of fermentation will yield benefits both to the processor and to the grower.

Growers and processors of sugarcane aided by milling and processing studies

The cooperative pilot-plant investigations on the milling and processing qualities of sugarcane, described in the 1951 report, were continued during a second grinding season at the Audubon Sugar Factory of Louisiana State University at Baton Rouge. Both plant and stubble canes were studied during the 1951 grinding season, to provide data on most of the new varieties studied. Samples of cane for these experiments were again supplied by the American Sugarcane League, the milling tests were carried out under a contract with the University, and processing experiments were conducted by the Southern Regional Research Laboratory.

About 2 tons of cane was used in each experiment. Results obtained during the two seasons were combined, and detailed analytical data on the composition of the juices and sirups were correlated with the pilot-plant processing results for both seasons. Publication of these data completes the first phase of the pilot-plant evaluation of milling and processing qualities of new varieties of sugarcane.

The principal purpose of this work was to obtain information on the milling and processing of new varieties of sugarcane so that the best varieties may be selected. Results of these studies of milling and processing behavior were considered recently by the Contact Committee of the American Sugarcane League, together with agronomic and other factors, in deciding on the release of one of the new varieties for commercial introduction. Although yields, sugar content, disease resistance, and other agronomic qualities of sugarcane are of primary importance, the pilot-plant processing results make it possible for processors to anticipate the processing behavior of a new variety, to adapt processing conditions to overcome difficulties, and to handle the cane efficiently.

An important element in making these results useful to the industry was the distribution of processing data to the entire industry within 2 months after the end of the grinding season. This information has been of value also to factories that produce only sirup rather than raw or refined sugar. One of the major purchasers of edible cane sirups has been furnished all the publications and some additional data from these experiments; the chemical compositions of the juices and sirups, as well as their clarities and sediment contents are being used as a guide in selecting better varieties for production of sirup.

Completion of this phase of the research, in which thorough pilot-plant processing evaluations were obtained 2 years prior to the possible release of new varieties, indicated the desirability of better testing and evaluation procedures that will be applicable to small samples of cane. Such small-scale tests would make it possible to evaluate new selections early in the breeding program. For this reason, a study is being planned to develop test methods applicable to as little as 25 to 50 pounds of cane.

The value of this work has been recognized in other sugar-producing countries, and requests have been received from them for details of pilot-plant facilities used in the processing work at the Southern Laboratory. To provide this information, a complete description of the plant and the arrangement of its units, together with working drawings from which the equipment may be constructed, has been prepared for publication.

Oilseeds

Global edible spread developed for military use

Although butter and margarine are used throughout the world by the Armed Forces, they spread easily only within a limited range of temperatures. At temperatures as low as 20° F. both are hard and difficult to spread, and at temperatures as high as 100° they melt. When either product is alternately cooled to 20° and warmed to 100°, it is likely to separate into two parts, one containing water and the other fat. Consequently, neither is suitable for military use where there are such extremes of temperature.

Spreads containing combinations of liquid vegetable oil, modified fat (monoglycerides), vitamins, flavoring, and surface-active agents have been developed at the Northern Regional Research Laboratory. These mixtures can be readily spread on bread at temperatures from 0° to 110° or higher. Many can be spread at temperatured range of more than 100°. The actual temperature range for a given mixture, however, depends on the composition of the oil and of the modified fat. A wide variety of flavorings soluble in fat are suitable for the spread. Investigations on the acceptability of various spreads are being conducted in cooperation with the Food and Container Institute of the Quartermaster Corps, and cooperative studies are planned on the stability of the spreads deemed acceptable. Pilot-plant tests will then be made to determine whether they can be produced commercially with a minimum of change in present industrial equipment. Studies on the properties of various mixtures are planned to determine their suitability for other military and civilian products.

Further progress made in stabilizing flavor of soybean oil

Substances responsible for off-flavors in aged soybean oil identified.—In cooperation with the Agricultural Experiment Station, University of Illinois, and with the University of Pittsburgh, the Northern Regional Research Laboratory has studied the flavor constituents of unstabilized soybean oil and soybean products. These substances have been identified as volatile carbonyl compounds, and a number of them have been isolated and characterized. In addition, it has been established that the reaction of linolenic acid produces some of the same substances formed during the aging of soybean oil. Unquestionably, this reaction forms carbonyl compounds that have characteristic painty and grassy flavors. It remains to be determined which of these compounds is specifically responsible for the characteristic flavors of soybean oil products.

The Illinois Experiment Station has carried on research on the substances formed from polymers of linolenic acid; the University of Pittsburgh has studied the products of reactions of isolinoleic acid—a constituent of soybean oil shortening; and the Northern Regional Research Laboratory has investigated the products of the reaction of linolenic acid and soybean oil. The projects at the University of Illinois and the University of Pittsburgh were carried on under research contracts.

Basic requirements established for metal inactivators for soybean oil.—Basic information on the chemical structure of substances that will inactivate metals in vegetable oils has now been obtained.

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Chemically, these metal-inactivating compounds must be able to react with the metal to form stable and inert compounds. Structurally, the metal-inactivating compound must have a reactive group that effects a primary reaction with the metal. These reactive groups are generally acidic, such as phosphoric, sulfuric, sulfonic, and carboxylic. The compound should also have another reactive group, which can stabilize the primary reaction to make the metal impurities inert. This information is of particular importance in the search for a nontoxic, heat-stable, fat-soluble, tasteless, and odorless inactivator for metallic impurities in fats. To eliminate ineffective compounds, the principles developed are being applied in selecting compounds for test purposes. This screening has led to the discovery of many new inactivators that are effective on a laboratory scale, including such compounds as iminodisuccinic acid, starch phosphate, and carboxymethylmercaptosuccinic acid. Studies on the toxicity, heat stability, ease of synthesis and cost, solubility, and flavor characteristics will determine which of the many compounds shauld be recommended for commercial trials. The same principles are applicable to all edible fats.

Procedure developed for adding gums to soybean oil to improve its flavor stability.—It has long been known that lecithin, extracted from crude soybean oil gums, is effective for stabilizing soybean oil against oxidation and deterioration of flavor. However, if lecithin in amounts sufficient to obtain stabilization is added to soybean oil immediately preceding the deodorization step, the oil develops a brown color. If it is added after the oil is deodorized, undesirable flavors may be introduced, and the oil darkens when it is heated to temperatures ordinarily used in cooking. It is understandable why commercial processors prefer to use citric acid or other stabilizers that are no more effective than lecithin but do not have its disadvantages.

Experiments conducted in stainless steel pilot-plant equipment demonstrated that the gums obtained from crude soybean oil may be returned to the oil between the refining and bleaching steps (to the extent of 0.1 percent by weight) to improve the stability of the oil and prevent darkening during the subsequent deodorization. In the pilot-plant studies, the crude gums were mixed with the refined oil for 15 minutes at 150° to 220° F. The bleaching agent was then added at 220°. The oil was bleached for 15 minutes, after which the bleaching agent and gums were filtered from the oil. When gums are added to the oil before it is bleached, the stabilizing effect is slightly less than when they are added between the bleaching and deodorizing steps. Adding the gums before the bleaching operation, however, stabilizes the flavor of soybean oil nearly as effectively as does citric acid. One of the advantages of this method is that the gums may be used just as they are obtained from the crude oil, whereas the gums must be treated to prepare the lecithin. Unlike citric acid and other metal inactivators, inexpensive gums are always readily available to the oil refiner.

Flavor of bread containing soy powder found acceptable

Nonfat soy powder contains highly nutritious vegetable protein. As "soy flour" it is available to the food industry for increasing the

nutritive value of bread and furnishing other desirable properties to doughnuts, crackers, and certain bakery products. All soy powders have characteristic flavors, which can be varied by proper selection of beans and by processing procedures. Most, if not all, of the raw-bean flavor can be eliminated by steam treatment of the flakes before they are reduced to powder. Any beany flavor remaining is practically eliminated when the bread is baked.

As a part of the studies on soy powder at the Northern Regional Research Laboratory, the effect of 5 percent of soy powder on the flavor of white bread was determined. The soy powder used was a composite mixture of four samples of commercial "soy flour" furnished to the Laboratory by individual companies. Previous studies had shown that uniformity within individual plants, as well as between plants, in producing soy powder had been improved enough to permit compositing. The composite soy powder was furnished to the pilot bakery of the Department of Milling Industry, Kansas State College, Manhattan, where bread was prepared in commercial equipment by the sponge-and-dough procedure.

Breads containing 5 percent soy powder or 4 percent nonfat dry milk solids were prepared in parallel runs and shipped overnight to the Northern Regional Research Laboratory. Flavor tests and other studies were made on breads from three different bakes. In studies designed only to determine whether these breads differed in flavor, more than 280 judges from the Northern Laboratory staff were unable to make a sufficient number of correct selections in 1,300 tests to indicate a significant difference between the breads. Only one person was able to make the correct selection in each of the six attempts. Apparently, only a small percentage of the population can detect a difference in flavor between breads containing 4 percent milk and 5 percent soy powder. It was not determined by the tests, however, whether the milk or soy powder contributed to this difference. The flavor of bread containing soy powder up to a 5-percent level should not be a factor in the acceptability of such bread by consumers.

New process extracts oil from cottonseed with solvents

A new process for extracting oil from cottonseed and other oilseeds with solvents was developed on a pilot-plant scale at the Southern Regional Research Laboratory. The process is called "filtration-extraction" because it uses a standard, horizontal, rotary vacuum filter as the principal operating unit. It is an efficient and simplified process for the direct solvent extraction of oil from cottonseed and, with certain modifications, other oleaginous materials such as soybeans, peanuts, flaxseed, rice bran, and tung kernels. Because it offers economic benefits over present commercial methods of processing, both mechanical and solvent, and because of its simplicity, the process is attracting interest for use in small and medium-size mills.

Filtration-extraction apparently overcomes most of the major shortcomings inherent in conventional direct extraction and in extraction preceded by prepressing. The costs for the equipment and operation are both relatively low, and the process produces high-quality oil and meal, as indicated by chemical analysis. It requires no radical or expensive departure from the operations now used to prepare cotton-

seed for hydraulic or screw pressing, although the method of preparation is modified. Equipment for the oil-extraction step requires relatively little floor space. For example, the pilot-plant filter being tested at the Southern Laboratory is only slightly larger than a family washing machine, but has a capacity of 24 tons of cottonseed a day, the approximate amount handled by a single commercial screw press in the same period of time.

In filtration-extraction, the prepared meals are mixed with miscella (a liquid derived from one stage of washing and consisting of about 10 percent oil and 90 percent hexane) in a slurry mixer-conveyor. After a contact time of 15 to 25 minutes, virtually all the oil is in solution in the form of a concentrated miscella. The slurry, with the admixed miscella, is then deposited continuously on a horizontal rotary vacuum filter, where the miscella is separated from meal particles by countercurrent displacement washing with commercial hexane. During the stay on the filter, about 1 or 2 minutes, the solids in the slurry receive three countercurrent washes, which successively reduce the residual oil content of the final meal to 1 percent or less. The concentrated miscella, removed at the first stage of filtration, has an oil content of approximately 30 percent, and contains only about 0.2 percent fines. It can be easily clarified, evaporated, and stripped for oil and solvent recovery in conventional equipment. The solvent-damp meal, containing only 20 to 25 percent solvent, is dried, and the solvent is recovered in conventional meal driers.

The high capacities attained with the filtration-extraction process, the excellent yield of high-quality oil, and a meal product with 0.03 percent or less of uncombined gossypol and high soluble-protein content (50–55 percent) are all dependent on the adequate rolling, moist cooking, and partial drying of the meals. These operations, however, can be easily carried out in conventional equipment used in hydraulic and screw-press mills with only minor additions and slight modifications. The proper conditions for rolling and cooking yield a granular material in which objectionable fines are consolidated into large agglomerates. This material has the desired characteristics for an efficient filtration-extraction operation, such as rapid extractability of the oil, particles of the optimum shape and size distribution, and relatively high incompressibility.

Feeding tests on the nutritive quality of cottonseed meals produced by filtration-extraction are being made by the California, Texas, and Louisiana Agricultural Experiment Stations, the Bureau of Animal Industry, and a manufacturer of feeds.

Cooperative agreements have been negotiated with mills and equipment manufacturers, and the filtration-extraction process is being operated and further evaluated on a full industrial scale. Demonstration of the commercial feasibility of the process will increase the already widespread interest in the conversion of the cottonseed-crushing industry from mechanical to solvent processes. Although solvent extraction was not applied to cottonseed in the United States until 1947, about 7 or 8 percent of the cottonseed was processed in five solvent-extraction plants during the 1950–1951 season. The number is expected to reach 22 in 1952–1953. These 22 plants probably will process at least 25 percent of the total cottonseed crop.

Grains

Protein-fat combination affected by other ingredients in bread doughs

The lipids (fats or fatlike constituents) in wheat flour affect the baking properties of doughs, but the mechanism that leads to these effects has never been explained satisfactorily. There is no doubt that basically the wheat proteins, rather than lipids, give doughs their ability to cohere, retain gas, and expand during fermentation and baking. Earlier work in the Western Regional Research Laboratory showed that when flour is mixed with water most of the lipids combine with the proteins in some unknown manner. Consequently, it appeared that this combination might affect the properties of the dough. This possibility is of particular interest in studies on the storage stability of wheat, flour, prepared mixes, and baked products. Lipid constituents of such products usually deteriorate more rapidly in storage than do other constituents, and added fats of poor quality affect the bread-baking properties of doughs adversely. Also the lipid-protein combination might explain some of the well-known variations in the baking properties of sound, fresh flours, which have not been explained by study of the protein components alone. This knowledge would have valuable applications in wheat breeding and processing.

As the first step in studying the lipid-protein combination (or binding of lipid to protein) that occurs during the mixing of dough, the effects of various ingredients, other than flour and water, on lipid binding were determined. Some ingredients, including yeast and sugar, had no effect; shortening agents and salt, however, showed interesting effects.

A common fat—lard—was not bound to the protein during the mixing of the dough and did not interfere appreciably with the binding of the flour lipids, which occurred to about the same extent as in the absence of lard. A bread "softener" of the polyoxyethylene stearate type (at present an ingredient not permitted by the Bread Standards) was not bound but actually decreased the binding of flour lipids. These results showed that the nature of lipids affects their combination with flour proteins.

Salt also decreased lipid binding. This result was of particular interest in studies of wheat gluten. Gluten, which contains most of the protein of flour, can be separated by kneading dough in a stream of water to wash away other constituents of flour. The finding that salt decreases lipid binding suggested that glutens washed out in water would contain more lipid than those washed out in a 2- or 2.5-percent salt solution. This was confirmed with a typical bread flour. The gluten washed out in water contained 9 percent lipid; that washed out in a salt solution contained only 5 percent lipid. The former represents the type of preparation usually made for gluten studies, but the latter more closely represents the gluten in bread doughs and therefore should be more suitable for studies on baking properties. Finally, the effects of salt and the softener on lipid binding varied among flours, indicating a difference in the nature of proteins in the different flours.

Much more investigation will be needed before the significance of these findings in relation to baking quality can be evaluated. They should prove useful, however, in calling attention to factors not considered in earlier work and in suggesting new approaches to problems in wheat processing.

White rice canned by new method has qualities of freshly cooked rice

To increase the popularity of rice for home and institutional use easily prepared rice products have been developed in recent years. Frozen rice, dry quick-cooking rice, and canned rice are in this category. The canning of rice has presented various problems. One of the principal difficulties has been the partial breakdown of structure and formation of an agglomerate of pasty kernels when the rice is retorted. For this reason, canners have used parboiled rice, which does not become pasty. Although canned parboiled rice has good texture, it has a slightly yellow color, and its flavor differs from that of white rice. The Western Regional Research Laboratory has developed a canned white rice that has the desirable qualities of whiteness, flavor, and grain separation associated with well-cooked rice. The process is applicable to both long- and short-grain varieties.

The most critical factor in canning white rice was found to be the moisture content, which should be kept at 55 to 60 percent for best results. This level is obtained if the rice is soaked to an equilibrium moisture content of about 30 percent and then boiled for 2 to 4 minutes, depending on the variety. It is packed in C-enamel cans, which are sealed under a 28-inch vacuum and retorted for 1 hour at 240° F. These conditions are adequate for the product packed in cans of the size customarily used for rice (300 x 407), but tests showed that retorting can be continued for as long as 2 hours without serious impairment of texture, flavor, or color.

The grains of rice canned by this process remain separate and white. The product is prepared for serving by adding a small amount of boiling water to the contents of the can, or by immersing the can in boiling water for a minute. The rice is then fluffy and pleasing to taste, and the flavor, as scored by a taste panel, is about equal to that of freshly cooked rice. After 6 months' storage at room temperature the rice showed no significant deterioration. The process opens the way for preparation of canned foods based on rice, such as turkey with rice and Spanish rice.

Research aids development and expansion of rice-bran-oil industry

The rice-bran-oil group of the Southern Regional Research Laboratory was given the Department of Agriculture Superior Service Award in May 1952 for its contribution to the development of the rice-bran-oil industry. Commercial production of this new domestic product is largely a result of the interest created by basic and applied technical information made available to industry by this group. Growth of the industry has been rapid—production jumped from a negligible amount in 1949 to 7,500,000 pounds in 1951. This output is only a fraction of the 50,000,000 pounds that could have been produced from the rice crop. Processing plants now under construction are expected to double the production capacity.

The work begun by the Southern Laboratory in 1947 provided almost all the information necessary to establish a rice-bran processing industry. Eleven technical publications have been published, and several other technical reports are now in press.

Initial investigations by the rice-bran-oil group demonstrated that oil low in free fatty acids could be produced from freshly milled rice bran by extraction with petroleum solvent and that it could be refined and bleached by the standard methods used for other soft, yellow oils. Studies of the composition and physical properties of the oils showed that a high-quality, stable salad oil could be made by refining, bleaching, and winterizing the oil. It was found also that the hydrogenated (hardened) oil makes shortenings comparable with those produced from cottonseed oil.

Other investigations determined optimum conditions for storing bran to minimize the formation of free fatty acids in the bran oil before its extraction and led to the development of a method for reducing the amount of "fines" in the miscella (oil-solvent mixture) during extraction. The variation of the fat content of rice bran in different varieties was established for eight varieties of rice grown in the Southwest.

Poultry and Animal Products

Research shows way to improve stability of lard oil

Lard oil is the limpid, clear oil expressed from lard after it has been "grained" at a suitable temperature. It is produced in both edible and inedible grades. Edible lard oil is used in a variety of ways, and the inedible oil has a number of important uses such as machine-shop lubricants and in textile processing and finishing. As in red oil, however, the presence of large amounts of polyunsaturated acid constituents frequently results in undesirable odor and color as well as gumminess and stickiness.

Recent research at the Eastern Regional Research Laboratory showed that an improved lard oil can be prepared by processing lard or grease that has been subjected to a slight selective hydrogenation. Experiments indicate that this hydrogenation can be carried to a point where the polyunsaturated acid content is 5 percent or less without any substantial reduction of the yield of lard oil or without measurable effect on the titer and pour point of the product. Further advantages and economies are possible by combining the use of slightly hydrogenated lard and grease with subsequent crystallization from solvents to separate the stearin.

Because of its lowered content of polyunsaturated components, lard oil prepared in this manner has improved color and odor stability. For the same reason, it can be further improved by addition of suitable antioxidants.

Hot-oil treatment of shell eggs alters their functional properties

A special process for treating shell eggs to increase their stability during storage has been investigated by the Western Regional Research Laboratory by means of a contract with Iowa State College. This work was done in cooperation with the Poultry Branch of the Production and Marketing Administration. The process, called thermostabilization, consists in heating eggs in oil at 130° to 136° F.

for 16 minutes. It was proposed by the Missouri Agricultural Experiment Station about 10 years ago. Their studies and others during the ensuing years have indicated that the candle grade after storage and certain other characteristics of the egg (for example, condition of the egg white) are favorably affected by the process.

If shell eggs treated in this manner are to be useful, their cooking or functional properties must also be adequate. A study was therefore undertaken to determine whether the treatment has any serious effect on the functional properties of eggs. The most noticeable adverse effect was the change in the whipping and angel-cake-making properties of the egg white. The whites of treated eggs required an undesirably long time for whipping, and angel cake made with them had an undesirably small volume. The treatment, however, had no effect on sponge cake, plain cake, or custard made with the eggs. These observations apply both to freshly treated eggs and to treated eggs held in storage as long as 9 months; hence, the effect of thermostabilization on the functional properties is irreversible.

In addition to the adverse effect on the whipping and angel-cake-making properties of the egg white, the treatment tended to make the white stick to the shell, resulting in a lower yield of egg white. It also increased the difficulty of separating the yolk and white. It is obvious that the acceptability of the thermostabilized eggs will depend on the importance attached to these defects by the consumer, and this question is being investigated by organizations skilled in the field of consumer acceptance. The research workers believe that the alterations of the functional properties are not serious enough to eliminate the process from consideration as a means of maintaining appearance and candle grade of shell eggs during storage.

Quality of cut-up frozen poultry protected by antioxidant coating

An important limiting factor in the keeping quality of poultry in frozen storage is the susceptibility of the fat to oxidation. Studies at the Western Regional Research Laboratory demonstrated that this susceptibility can be decreased markedly by applying coatings that contain antioxidants to the surfaces of processed poultry. Such antioxidant coatings can be applied advantageously where large surfaces are exposed, as in eviscerated and cut-up poultry. The protective effect might be specially useful if packaging and storage are less than optimum as a result, for example, of economic or military conditions. Because turkeys are more susceptible to oxidative rancidity than chickens, and because seasonal production requires that they be stored longer, turkeys rather than other poultry were used in the initial studies.

Development of the antioxidant coating involved determination of the best antioxidant or combination of antioxidants, selection of a suitable solvent or carrier, and establishment of optimum concentrations of the antioxidants in the carrier. Only antioxidants that had been approved for use in food were considered. Marked beneficial effects were obtained with combinations of butylated hydroxy anisole, propyl gallate, and citric acid. Propylene glycol, coconut oil, hydrogenated vegetable oils, and aqueous gelatin were tested as carriers. An aqueous solution containing 3 percent of edible gelatin gave the best results.

Half turkeys, prepared by splitting eviscerated turkeys through the back and keel bones, and turkey steaks (⅝-inch-thick transverse slices from the frozen eviscerated carcass) were used in evaluating the antioxidant coatings. The frozen halves and steaks were dipped in aqueous solutions containing 3 percent of gelatin and various antioxidants dissolved or suspended in the aqueous gelatin. The coated pieces were packaged (steaks in sealed cellophane bags, turkey halves in a butcher-wrap) and stored at +10° F. for 6 months. The protection given by the various coatings, as compared with uncoated controls, was measured in terms of peroxides in the raw fat of skin and meat and rancid off-flavors and odors in the cooked skin and meat. Estimates of protection based on chemical analyses for peroxides agreed significantly with estimates made from subjective ratings for rancid off-flavor and off-odor by a trained taste panel.

The skin of the stored turkey steaks coated with aqueous gelatin without antioxidant had 30 percent less peroxide and 20 percent less of the rancid off-flavor than the controls. Steaks coated with aqueous gelatin to which antioxidants were added (0.1 percent butylated hydroxy anisole, 0.03 percent propyl gallate, and 0.02 percent citric acid) had 80 percent less peroxide than the uncoated controls and 50 percent less off-flavor. Similar results were obtained with light and dark meat. Concentrations of antioxidants one-tenth of those listed gave less than optimal results.

The aqueous gelatin coating that contained the antioxidants used on steaks but at one-fifth the concentrations listed had substantial protective effects on the half turkeys. There was 70 percent less peroxide and 40 percent less off-flavor. As for the steaks, the effect of the gelatin coating alone was intermediate. The general agreement of the beneficial effects on steaks and on half turkeys indicates that the methods should be applicable to any of the forms in which turkeys are marketed.

Foods—General

Unique facilities available for studying effects of temperature variations on frozen foods

One of the most important problems in the field of frozen foods is the effects of temperature changes on the quality of the products as they move through channels of production, storage, distribution, and retail sale. Many of the temperature changes can be avoided or minimized, and adequate knowledge of their effects on the quality of the product will be helpful in avoiding injurious temperatures.

Large-scale facilities for studying the effects of temperature on frozen foods have been designed and installed at the Western Regional Research Laboratory. To obtain statistically sound data, large quantities of frozen food samples must be exposed in the range of −30° to +40° F., with identical conditions of temperature and air movement for the packages. Heretofore, adequate facilities for such studies have not been available.

The unique new system consists of eight rooms in which samples can be stored under carefully controlled variable temperatures, two rooms in which samples can be stored under carefully controlled low temperatures, a refrigeration machinery room, and a main control panel board.

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In each of the variable-temperature storage rooms, blowers, duct work, and a combination heating and cooling coil unit are arranged to discharge air of controlled temperature through shelving located along three sides of the room. The temperatures of the rooms are automatically programed and controlled by instruments on the main control panel board. The same "refrigerant" is used to heat and cool the rooms. During the cooling portion of the temperature cycle, the refrigeration equipment in the room functions in the usual manner to cool the samples. During the heating part of the cycle, the same equipment and the same "refrigerant" are used to heat the samples by extraction of the required heat from other rooms, which are being cooled.

Limited work on fruits has been under way for about 2 years. Research on the temperature tolerances of vegetables and poultry was started in 1952.

Rapid analytical methods developed for use in food processing

Oil content of potato chips.—Oil constitutes approximately 40 percent of the weight of potato chips. The oil content varies with the potato, as well as with the method of preparing the chips. Methods available for measuring the oil have required so much time and equipment that investigations on this important constituent have been seriously impeded. In addition, the solvents used have constituted a serious fire hazard. A new method developed at the Western Regional Research Laboratory has reduced the time required for analysis from 20 hours to 25 minutes.

A representative sample of potato chips is crushed in a large mortar. A weighed sample is transferred to a sintered-glass crucible, and the oil is extracted with hot carbon tetrachloride. The solvent is evaporated with an infrared lamp, and residual oil is cooled and weighed. Several samples can be analyzed simultaneously.

Relatively simple equipment is used, and because of the simplicity of the technique, a minimum of skill is required. Carbon tetrachloride, the solvent used, is not inflammable.

The method is accurate enough for use in research laboratories, and it is so rapid and simple that it is being used in the control laboratories of potato chip processing plants.

Moisture content of army ration pineapple rice pudding.—The Western Laboratory was requested by the Army Quartermaster Inspection Division, Oakland, Calif., to recommend a rapid, precise method for determining moisture in pineapple rice pudding, a field ration item. The military specification called for 68.0 ± 0.5 percent moisture, as measured by an 18-hour vacuum-oven method. A moisture method capable of yielding equivalent results in minutes was required for controlling the manufacturing process, since the product must be canned immediately after it is prepared. The method for rapid measurement of water content of fruits and vegetables described in the 1951 report was found to be well suited to this purpose. The nonaqueous part of the sample is measured by the potassium dichromate required to oxidize it, and the water content is then determined by difference. Since the pudding contains pineapple, rice, eggs, milk, sugar, lemons, and starch, each of which may vary within specification tolerances from batch to batch, the effect of such variance on the mois-

ture content was determined. Values obtained by this method on a large number of batches were compared with those obtained by the vacuum-oven method. A mean difference of 0.11 percent of moisture (maximum difference, 0.30 percent) was found. The method has been successfully used for both specification and control purposes.

Feeds—General

Methods developed for recovery of tomato-processing wastes

Nearly 3 million tons of tomatoes are processed in this country annually. Since most of them are made into juice or juice products, almost 20 percent of the tomato solids are wastes. Disposal of this large amount of organic material is a serious problem. Only about 5 percent of the waste solids from tomato processing is recovered, and the few plants that recover the waste dry only the part that can be pressed; the fluid fractions are discarded.

In large-scale pilot-plant research at the Eastern Regional Research Laboratory, a process was developed by which all the press cake solids can be recovered as well as 65 percent of the solids in the waste liquors; 83 percent of the total waste solids is recovered.

In this process, culls and trimmings are chopped, and then passed through a finisher. The tailings from the finisher are combined with those from the juice-making operations and pressed in a continuous, screw-type press, yielding a cake and a liquor. The cake is dried in a direct-fired, rotary, alfalfa-type drier with inlet and exit gas temperatures of about 1050° and 275° F., respectively. These temperatures were satisfactory for press cakes with moisture contents of about 60 to 69 percent. The dried product had a good odor and light color and contained about 8 percent moisture.

About 65 percent of the combined liquors from the finisher and press, containing about 5 percent solids, is concentrated to about 30 percent solids in a submerged combustion evaporator. The dried press cake and concentrate are thoroughly mixed in a continuous mixer-conveyor, and dried in the drier used for drying the press cake. Chiefly because of sugars and other soluble substances in the mixture, it is necessary to use relatively low inlet and exit air temperature for drying this mixture. A satisfactory product is obtained with inlet and exit temperatures of about 550° and 235° F., respectively. Incorporating the concentrate from all waste liquors gave a sticky mixture that could not be dried to a satisfactory product.

In cooperation with the Delaware Agricultural Experiment Station, feeding tests with broilers are being made to determine the feed value of both the dried press cake and the dried mixture of press cake and concentrate when incorporated in broiler rations. Use of the dried mixture in dog rations is being investigated under a memorandum of understanding between this Bureau, the Agricultural Experiment Station at Cornell University, and the Sea Board Supply Co., Inc., of Philadelphia, Pa. The dried press cake is now used chiefly in dog rations.

The capital costs of a plant processing the wastes from a juice factory handling 800 tons of tomatoes daily are estimated to be about $113,000. This plant would produce 8.13 tons of feed each day during

the peak of the season at a cost of $116 per ton. In deriving this figure, not only were factory costs and overhead considered but interest on working capital and administration and general expense as well.

The value of the dried mixture of press cake and concentrate cannot be stated pending conclusion of current feeding tests with broilers and dogs. It is apparent, however, that in poultry rations the product is at least equal to wheat middlings. Moreover, when dumping is no longer possible, the recovery procedures suggested here may well represent the cheapest means of disposal.

Studies on toxicity of trichloroethylene-extracted soybean-oil meal

From 1948 to 1952 numerous outbreaks of a hemorrhagic disease in cattle fed trichloroethylene-extracted soybean-oil meal (TESOM) occurred in the United States. These outbreaks were associated with meal produced primarily in new plants using modified processing procedures. Although previous outbreaks of a similar disease had occurred in England in 1912, and in Germany and the Low Countries in 1923–24, oilseed-processing plants in England and the United States had operated from 1939 to 1947 without any reported cases of this disease. Work undertaken at the Iowa, Minnesota, Kansas, and North Dakota Agricultural Experiment Stations has established that the recent outbreaks of hemorrhagic disease in the United States are similar to those in Europe and that the causative agent, not yet determined or identified, is present in trichloroethylene-extracted soybean-oil meal.

Extraction of oilseeds and fat-containing materials with trichloroethylene, which is relatively noninflammable, does not present the explosion and fire hazards that accompany the use of highly inflammable solvents. For this reason, primarily, trichloroethylene has been the preferred solvent in a number of small solvent-extraction plants that process soybeans. When the toxicity of TESOM was recognized, four large plants for extracting soybeans with trichloroethylene, then under construction, were converted for use of the petroleum hydrocarbon hexane, as the solvent.

The Bureau has undertaken research on the toxicity of TESOM, with the immediate goal of developing suitable tests for detecting such toxicity, to isolate and identify the toxic factor, and to assay quantitatively the toxicity to young cattle, swine, and poultry. This work is being done at the Northern Regional Research Laboratory, and under research contracts with Iowa State College and the University of Minnesota, at the respective agricultural experiment stations. The final goal of this work is to find a simple unequivocal test for the toxic factor and to determine what processing conditions do or do not cause it. This information is essential to any successful resumption of commercial extraction of soybeans with trichloroethylene. Preliminary results indicate that toxicity may be associated with high temperatures and changes in the solvent during the processing of the soybeans.

Wet process for dividing sugarcane bagasse into pith and fiber improved by pilot-plant studies

The importance of the wet-separation process for dividing sugarcane bagasse into pith and fiber proposed by the Northern Regional Research Laboratory and discussed briefly in the 1951 report is well

illustrated by a recent statement of the paper and feed manufacturing industries.

"The key to successful use of bagasse for the production of pulp is an efficient and successful means of separating pith from the fiber, followed by some economic use of the pith. The direct farm feed consumption market (for blackstrap molasses) is the largest in potential and smallest in actual present annual sales volume. * * * This market is not attainable until the problem of how to get molasses in dry form at reasonable price to the farmer is solved. The two types of solution to this problem are: (*a*) dehydration of molasses to a granular nonhygroscopic form, (*b*) absorption of molasses on a porous feed material to produce a dry, granular, nonhygroscopic product containing 80 percent or more of molasses."

This new process has been intensively studied on a small pilot-plant scale. The pith is first loosened from the fiber at high concentrations in water without appreciably cutting the fiber or breaking down the pith particles to destroy their absorptive properties. A study of two commercially available machines for carrying out this step has been completed. One operates batchwise; the other, continuously. The latter operates at much higher capacity. It also requires less floor space, less power, and lower capital investment. The loosened pith is separated from the fiber by screening. Although this operation is different from the screening of paper pulps, pulp mill screens of two types have been found that operate within a practical range. To obtain a highly absorptive pith, the pith fraction separated by one screen must be passed through another screen to remove coarse fibers. Both the bagasse fiber and the pith are dewatered before drying. The fiber may be dewatered to 45 percent water content by running it through sugar mill rolls. Pith with a moisture content of about 6 percent can be collected from the pith slurry by use of either a vibrating screen or a rotary vacuum filter. Tests with a commercial vibrating screen showed that loss of pith through the vibrating screen is too high for such a screen to be practical. The pith slurry is easily filtered, however, and a good cake is formed on a vacuum filter. More data are required on this step, as well as on the dewatering of the pith before drying. Preliminary tests with a commercial dewatering press indicated that pith containing 60 percent water can be obtained. Both the pith and the fiber are dried. These studies have not yet been completed.

Studies have been directed to finding a quick, simple method for determining the relative absorbency of pith. Although this proved to be rather a difficult problem, a method was developed that gives reproducible results. This method, together with another for fractionating the pith slurry to determine the percentage and character of the fiber, has materially assisted in the progress of these studies.

It has been found that mixtures of 30 parts pith and 70 parts blackstrap molasses are nonhygroscopic and do not cake when stored. With highly absorptive pith, pellets can be made of 15 percent pith and 85 percent blackstrap molasses. Several producers of mixed feeds are interested in the absorptive properties of pith.

The Hawaiian Sugar Planters Association is cooperating in these studies under a formal memorandum of agreement; three collaborators are working at the Northern Regional Research Laboratory. Sugar

companies in Louisiana and Florida have cooperated in supplying large samples of various bagasse varieties and in making dry-screening and caking tests. Cooperation is also being arranged with mills in Puerto Rico.

Bureau method for production of vitamin B_{12} used by industry

Phenomenal benefits of vitamin B_{12} in animal nutrition led to its production by fermentation to meet steadily increasing demands. A process developed at the Northern Regional Research Laboratory for producing vitamin B_{12} by a micro-organism, *Streptomyces olivaceous*, was described in the 1951 report. This process has been adopted by six industrial firms, which manufactured upward of 25 percent of the vitamin used in livestock feeds during the past year. Laboratory tests showed that *S. olivaceous* also produces other vitamins, such as riboflavin, niacin, pantothenic acid, biotin, and pyridoxine, together with an antibiotic designated "olivacin." The fermentation product is much more potent in B_{12} than in any of the five other vitamins, but the others are of benefit. In feeding tests with chicks, conducted by the Michigan State Agricultural Experiment Station under a research contract, the growth-promoting activity of vitamin B_{12} produced by *S. olivaceous* was confirmed and that of the accompanying antibiotic substance was determined.

When incorporated in a plant protein ration, the fermentation product of *S. olivaceous* containing a given amount of vitamin B_{12} was considerably more effective in promoting growth of the chicks than was the same amount of the pure vitamin. The appreciable growth-promoting effect of olivacin thus indicated was later confirmed in feeding tests with a partly purified preparation of the antibiotic. Chemical and storage tests showed, however, that the antibiotic is relatively unstable, and it is not recommended as a growth-promoting adjunct in feed supplements. Vitamin B_{12}, on the other hand, remains stable after storage for at least 2 years, and the new process developed by the Bureau produces it in sufficient concentration to warrant its manufacture as a primary product.

Fruit cannery wastes processed into valuable products

In cooperation with the Canners League of California, research phases have been completed on a practical process for the large-scale utilization of fruit cannery wastes. Results of two seasons (1949 and 1950) of pilot-plant studies in facilities owned by a group of fruit canners in the San Jose, Calif., area were described in previous reports.

During the 1951 season, the pilot plant was operated as a small commercial plant, processing about 150 tons of raw pear waste in 24 hours. The major change from previous seasons was the use of the new, continuous waste treater, completing conversion of the plant from batch to continuous operation.

The new waste treater performed at or above its rated capacity throughout the season and gave every indication of being a thoroughly practical unit for a commercial utilization plant. Chemical treatment of the waste when applied in the continuous treater facilitated separation of juice even more effectively than during the previous season. The improvement due to the continuous treater resulted in a 50-percent increase in the capacity of the dejuicing press.

During much of the 1951 season, the utilization pilot plant was operated on two shifts a day. A throughput rate corresponding to 150 tons a day was maintained for many days, and was frequently exceeded for several hours. The plant processed about 1,800 tons of pear cannery waste during the season and produced 145 tons of pear molasses and nearly 75 tons of dried pear pomace. An equivalent amount of wet press cake was hauled to a disposal area because of inadequate drying capacity at the plant. The products, shipped in bulk from the plant, were readily sold for feeds, bringing $45 a ton for molasses and $30 a ton for dried pomace.

The season's work demonstrated that the process developed for utilizing pear cannery waste is practical and economically sound. The methods developed for conversion of pear waste to useful products—molasses and pomace—are applicable to other fruit-processing wastes without major change. After the 1951 season, the pilot plant was sold to a corporation organized for the purpose of processing fruit and vegetable wastes.

Feeding tests carried out by the California Agricultural Experiment Station indicated that both the pear pomace and molasses produced in the San Jose plant were suitable for use in mixed feeds for cattle and sheep, and that the pear molasses was superior to cane molasses in feeding value.

The pear pomace was carefully tested to determine possible toxicity from spray residues. A typical pear pomace produced from cannery waste may contain lead, arsenic, fluorine in the form of cryolite, a chlorinated hydrocarbon such as DDT, and an organic phosphate such as parathion. Individual spray residues have been studied, and tolerance limits or suggested tolerance limits have been established for the maximum amounts permissible in feeds, but mixtures of the residues had not been tested previously for additive or synergistic effects. A control pomace was made from pears obtained in an unsprayed orchard; comparable pomaces were prepared from pears produced in orchards where a controlled spray program was used. On the basis of analyses of the lead, arsenic, fluorine, and DDT contents of pomaces, a composite pomace was prepared containing each of the spray residues in a concentration at least as high as that which would occur under normal conditions. Weanling albino rats were placed on a basic diet; others were fed the same diet and in addition 10, 20, and 30 percent, respectively, of the control pomace or the composite pomace. No evidence of toxicity attributable to the insecticidal residues was noted, as judged by growth rates, blood morphology, color of incisor teeth, and gross and microscopic examination of tissues.

The concluding paragraph of a statement released by the Canners League of California contains an appraisal of the pear waste project by the industry. "As a direct result of the research program of the past 3 years, California fruit canners now believe they have a sound alternative to present waste disposal methods. Cooperation of industry and Government has served to bring this project to a highly satisfactory conclusion, which is of great importance both to the canning industry and to the public."

Sugar-beet-top feed value conserved by dehydration

The lush green foliage of a field of sugar beets indicates the high feed value of the leaves of this crop. The yield of dry matter from

the leaves and stems approximates that of a cutting of alfalfa, and the protein and carotene contents also are comparable with those of alfalfa. In some growing areas, the tops are pastured after the roots are hand-harvested; in others they are field-cured, made into small cocks, and hauled to the feed lot. In still other areas, the tops are left in the field and plowed under for their soil-conditioning value. These methods of utilizing the tops for livestock feed are wasteful. Fifty percent of the dry matter, sixty percent of the carotene, and fifty percent of the protein are lost during 10 days of field curing. At present rates of production of sugar beets, properly harvested tops would yield about a million tons of high-grade forage.

Under a research contract supervised by the Western Regional Research Laboratory, the Colorado Agricultural Experiment Station has completed a study on the dehydration of sugar-beet tops as an economic means for preserving their nutrient values. Four ways of handling the tops were investigated: (1) Reduction of moisture by pressing before dehydration, (2) reduction of moisture by ensiling before dehydration, (3) dehydration of a mixture of chopped leaves and pressed beet pulp, and (4) direct dehydration in a commercial-type alfalfa drier.

The direct pressing of chopped tops was not found feasible because the commercial continuous presses and experimental presses used expressed too high a proportion of pulpy material with the juice. A large reduction of moisture was accomplished by blanching and hydraulic pressing, with little loss of protein and carotene. The resulting press cake was dehydrated readily into an excellent meal. Ensiling has the advantage of reducing moisture without appreciable loss of nutrients, and also it is a means of stockpiling the tops for later drying. Experiments on ensiling with acid showed that such treatment is economical, provides an excellent way of prolonging the dehydrating season, and produces a dehydrated meal of high nutritional value. A mixture of pressed beet pulp and chopped leaves dried in a pulp drier showed some separation during the drying and handling cycle. This equipment, however, could be adapted to do a satisfactory job. Addition of beet tops to the pulp resulted in a product of higher protein and carotene content than pulp alone. Direct dehydration of both chopped tops and chopped leaves in commercial alfalfa dehydrators was successful. Leafy material dehydrated in this manner had a dark-green color, a protein content of about 15 percent, and a carotene content of about 100,000 I. U. per pound. Extensive feeding experiments showed that in feed value and palatability dehydrated beet tops are equivalent to dehydrated alfalfa with comparable protein and carotene contents.

Studies indicated a production cost of $42.84 a ton for dehydrated beet tops and $50.70 a ton for dehydrated beet leaves. These costs include payments to the grower of $5.21 and $11 a ton of dry material for the tops and the leaves, respectively. A complete report of this study is being published by the Colorado Agricultural Experiment Station. It will bring together information on the various phases of sugar-beet-top processing and utilization. These data will enable the grower or processor to choose a method of handling best suited to his particular needs.

FIBERS

Cotton

New draft guides lower cost of making cotton yarns

Spinners of fine-cotton yarns have reported that the new draft proportions developed at the Southern Regional Research Laboratory to replace the manufacturer's guide for a long-draft roving system helped them to produce substantially more uniform yarns and lowered their processing costs. Draft proportionment between the front and rear zones is a critical factor in obtaining maximum uniformity of roving on long-draft frames, used by almost all mills, but with the machinery manufacturer's draft guides the machines do not always deliver roving of the greatest uniformity of which they are capable. Roving of maximum uniformity is highly desirable because of the direct relation between uniformity of roving and quality of yarn.

Studies of drafting long fine-fibered cotton, which led to this response by the fine-yard spinners, were described in the 1948 report. Similar studies have now been completed for a thirty-one thirty-second-inch coarse-fibered cotton, representative of cottons ranging from seven-eighths to 1 inch in length. Proper ratios of draft between the rear zone and the total draft, and the proper ratios of the weight of stock fed to the total draft required for maximum roving uniformity have been determined for the three principal long-draft roving systems. Tests showed that better quality rovings and yarns and more efficient machine performance were obtained with the new draft guides. Roving uniformity was 5 to 10 percent better, and the variability of the weight-per-unit length and strength of the yarn was about 10 percent lower.

Work is in progress to develop draft guides for these three long-draft systems with cottons of medium length and fineness. Data on these typical cottons should provide the proper draft proportions to obtain roving of maximum uniformity from any American cotton. Although the dollar value of such studies is difficult to assess, they will unquestionably lead to substantial economies because of improved quality of cotton products, improved machine performance, and reduction in waste.

Improved cotton fabrics made with new loom attachment

The reports for 1949 and 1950 mentioned that a loom attachment was being developed at the Southern Regional Research Laboratory that would enable conventional looms to weave abnormally dense cotton fabrics. This study was undertaken primarily because of the interest of the Army Quartermaster Corps in fabrics of this type. Since release of the plans for an improved model in June 1951, engineering drawings for construction of the device and instructions for its operation have been furnished to representatives of almost 500 textile mills. The attachment extends the weaving range of a loom well beyond the maximum number of picks per inch normally obtainable. The tightly woven fabrics are so highly resistant to water and wind that they do not require chemical treatment. The attachment uses a number of standard loom parts, and can be installed on most looms at a total cost of less than $100. It is simple to adjust and use.

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In addition to producing abnormally dense cotton fabrics, the new attachment improves the quality of many types of standard fabrics with the normal amount of pickage. It reduces the number of reed marks—thick and thin places—thereby increasing the smoothness of the fabric. Also, it makes standard fabrics less permeable to air and more resistant to tearing, and gives them greater strength in the direction of the warp. These improvements should increase the utilization of cotton.

Expanding use of cotton opener brings savings to textile mills

The 1951 report described the first use by textile mills of the lint cotton opener developed by the Southern Regional Research Laboratory. During the past year, use of the new opener has increased. Ten of the machines are now operating in seven textile mills, and it is known that orders have been placed for a number of others from the eight manufacturers licensed under Government patents to produce them. Industry spokesmen have expressed the opinion that this cotton opener is one of the outstanding developments in textile equipment of the past quarter century.

The merit of the opener has been proved under commercial conditions. Full-scale production confirmed laboratory findings that the opener did not cause any fiber damage and that it enabled better cleaning of the lint, with less spinnable fiber being removed with the trash. The published report of one mill on the processing of 6½ million pounds of cotton through the opener said: "The lint cotton opener developed at the Southern Regional Research Laboratory is doing an amazing job. Total waste has been reduced. Yarn quality is definitely improved." This mill's detailed records showed an overall reduction of 1 percent in waste, 22 percent less spinnable fiber lost in the opener-picker waste, a much cleaner picker lap, better blending, no increase in neps, 5-percent increase in the strength of the yarn, and approximately one-half grade increase in the appearance of the yarn. Savings by this mill, which processes 40,000 bales a year, are estimated at $2 per bale. This amounts to $80,000 annually, a sum approximately equal to the total expenditure for research to develop the opener.

The potential value of the opener can be estimated from the 1951 crop, of which virtually one-half was graded Strict Low Middling, or lower. If this low-grade cotton had been processed through the opener with a net saving of only $1 a bale, over-all manufacturing costs would have been reduced by $7.5 million, and the machine-harvested and other low-grade cottons would have been more adequately and efficiently cleaned for quality textile products.

New tests for cotton textiles aid to research and production

For more careful selection of cottons for different uses and development of new and improved textile products, improved testing methods and equipment are needed. It is believed that the two following methods developed by the Southern Regional Research Laboratory will be of value to both research and industrial agencies.

Rapid test indicates field damage.—The degree of alkalinity of cotton provides an index of the deterioration caused by field weathering under humid conditions, which may prevail from the time the bolls open until the cotton is picked. The water extract of a weathered

cotton is usually alkaline. The alkalinity is produced by the action of micro-organisms, which may weaken the fibers and alter other physical properties to such an extent that the raw stock is undesirable for processing into yarns, cords, and fabrics.

To provide a rapid test for detecting field damage, a colorimetric method was developed for determining the acidity or alkalinity of cotton fiber. The results are expressed in units of pH. For example, cotton having a pH of 9 is strongly alkaline and likely to make low-quality yarns and fabrics.

In this test, neutralized sulfonaphthalein indicators in 25-percent isopropyl alcohol are applied directly to the fiber. The pH value of the fiber is then determined by comparison with color standards which consist of cottons that contain an indicator solution and appropriate amounts of an acid or an alkali. The test can be made in approximately 2 minutes. The results closely approximate the actual pH value of cotton fiber, and are in good agreement with those obtained by precise, time-consuming techniques. The new method should be useful to cotton mills as a screening test in sorting cottons.

NEW APPARATUS DEVELOPED FOR EVALUATING THE WARMTH OF TEXTILES.—A new apparatus for measuring the warmth of cotton and other fabrics promises to be useful commercially as well as in research to develop improved cotton products. The equipment was designed at the Southern Regional Research Laboratory to fill a specific need in evaluating the warmth of experimental cotton fabrics, but it has attracted the attention of other laboratories and of industry as well.

Although warmth is an important service requirement of many textile fabrics, it is difficult to evaluate and, in spite of the fact that many methods have been developed by various laboratories, there is no generally accepted test. In most of these methods the warmth value of a fabric is the difference in energy required to maintain a metallic object at constant temperature in still cold air when uncovered and when covered with the test material. The apparatus developed at the Southern Laboratory applies this principle, but instead of being tested in still air the fabrics are subjected to a simulated wind along with low temperatures.

A metal "arm" clothed in a sleeve of the test fabric is lowered into a commercially made home freezer in which a fan has been installed to maintain a wind velocity of 12 to 13 miles an hour over the surface of the fabric. This combination of wind with cold temperatures, which can be adjusted as desired, provides a better evaluation of fabrics under conditions similar to those prevailing in Arctic regions and other cold climates than is provided by the more usual tests in still air.

The warmth-testing apparatus has been used to evaluate a number of experimental cotton fabrics and to compare them with cotton and other commercial fabrics.

Carding process for cotton mills improved

Methods for reducing the formation of neps during cotton textile processing, developed by the North Carolina State College of Agriculture and Engineering under a research contract with the Bureau, were described in the 1951 annual report. The project, now completed, has produced additional improvements in mechanical processing. One

phase of the investigation showed that the speed of the card lickerin is the key to decreased neps, increased production, and lower costs in the carding process. As compared with a normal lickerin speed of 430 revolutions per minute, a speed of 800 revolutions per minute makes possible increases in carding rates from 60 to 100 percent on cottons of 1 inch or less without adversely affecting formation of neps or the quality of the sliver. This increase will materially assist in overcoming the bottleneck in carding that always arises when the textile industry is called upon to produce large quantities of cotton goods for defense purposes. It will also enable mill operators to balance the production of cards with the production of other machines in the mill. This is important because new cards are unavailable in times of national emergency.

A number of textile mills have put these research findings into effect. One mill reports they have "* * * increased their carding rate from 15.7 to 25.7 pounds per hour, carding costs are down, breaking strength and sliver variation are improved, with fewer neps * * *." They estimate that the 62 percent higher production, with less waste removed at the card, saves about $680 per card per year. It is estimated that more than a quarter of a million dollars a year would be saved by a mill operating 370 cards at this increased rate. This saving is more than four times as much as the cost of the investigation that led to the improvement.

Cotton made more wrinkle-resistant by new two-step process

More than 2 million bales of cotton are now consumed annually in clothing and household fabrics, in which crease resistance is desirable. Commercial methods of achieving creaseproofness in cotton and other cellulosic textiles are based on the use of resins. Although each year millions of yards of cotton goods are treated with resins to impart resistance to wrinkling, much better methods are needed to improve this quality in cotton products.

Because resistance to wrinkling is important to the consumer, this property has been studied at the Southern Regional Research Laboratory. The difficulty in obtaining optimum creaseproofness in cotton cloth commercially apparently stems from imperfect distribution of the creaseproofing material in the fiber. Because of its structure, untreated cotton resists penetration. In a new approach to the problem, the Southern Laboratory developed a method that improves wrinkle resistance by increasing the absorption of the fiber. It utilizes the usual creaseproofing resins but applies them to chemically modified cotton rather than to plain cotton. The cotton is altered to provide an acid group chemically attached to the fiber. Several processes are capable of effecting this change, but carboxymethylation was found most suitable. It consists in impregnating the cloth with monochloroacetic acid—a low-cost material—and then treating it with a strong solution of sodium hydroxide, causing the acid to unite with the cellulose. The increased swellability of carboxymethylated cotton permits the resins to penetrate the fibers easily when applied in the usual manner. The resulting product has the desired pleasant feel, better resistance to abrasion, and the usual excellent properties of cotton.

An additional benefit is that the acidity of the carboxymethylated group provides for effective condensation of the resins, without addi-

tion of the mineral acid salts usually needed for this purpose in commercial processes. The possibility of degradation of the cotton by mineral acids is thus eliminated.

Several companies have shown interest in carboxymethylating cotton to obtain superior crease resistance. Production costs should be moderate, because carboxymethylation can be carried out with ordinary textile equipment, and a small amount of monochloroacetic acid is sufficient to obtain the reaction to an effective degree. A public service patent has been issued on the process.

Phosphorylated cotton—another chemically modified material—can also be used to obtain good crease resistance, and other modified cottons may prove suitable. This work on creaseproofing emphasizes the many avenues of improvement opened up by research on the chemical modification of cotton.

New, body-conforming cotton bandage produced commercially for the armed services

A new type of cotton gauze bandage of superior elasticity and clinging power, developed by the Southern Regional Research Laboratory, was reported in 1943 and 1944. Thousands of rolls of this bandage were produced experimentally, and tested by Army and Navy surgeons during World War II. During the present emergency, the Office of the Surgeon General has conducted additional tests in Army hospitals and in the field in Korea. Success in these trials was responsible for moving the new bandage into commercial production.

The new product is made by a special method for shrinking ordinary cotton gauze in strong caustic soda solution. This mercerizing treatment causes the fibers to swell, and in swelling the yarns crimp and kink. The resulting elasticity of the gauze in both directions makes the bandage almost incapable of slipping. It fits the joints snugly, allowing elbow, knee, wrist, or ankle more freedom to move than do ordinary bandages. The pronounced elasticity of the bandage gives it a self-tightening quality especially suitable for mild pressure dressings for burns and skin grafts. This true elasticity, too, allows not only movement in a bandaged joint but flexing through a wide angle without loosening the bandage. The tendency of successive layers of the gauze to cling together produces the nonslipping property.

This body-conforming bandage was the best suited of any tested for securing the new universal protective dressing developed by the National Research Council and Office of the Surgeon General for large burned and multiple-wound areas. A still further improvement made at the Southern Laboratory was the use of unbleached gauze instead of the usual bleached surgical gauze formerly used. One firm is now making the bandage, and another is expected to start production during 1952. The new bandage can be furnished at a cost less than half the cost of the conforming bandage previously used. The Armed Forces have saved several millions of dollars on the initial procurement order.

New compounds promise more satisfactory flameproofing of military clothing

An extensive program of chemical research to develop better flameproof cotton fabrics has been undertaken by the Southern Regional Research Laboratory at the recommendation of the Army Quarter-

master Corps. Practical reasonable-cost methods are being sought for making fabrics permanently flameproof without imparting undesirable properties. Such cotton fabrics are needed greatly by the Army for clothing and tentage and by civilians for many purposes, such as clothing, draperies, curtains, and other household articles. New types of incendiaries—flame throwers, napalm bombs, and other forms of fire—have created a demand for fabrics with improved flameproof properties.

Although many flameproofing treatments have been developed by different research organizations, none has proved entirely satisfactory for military use. The ideal flameproofing treatment for clothing must make the fabric permanently flameproof, and also glowproof. The treatment must not weaken the fabric, nor should it make the fabric stiff and boardlike, uncomfortable to wear, or harmful to the skin. From the standpoint of commercial processing, it is desirable that the chemical be applied in a water-dispersion rather than in an organic solvent.

The Southern Laboratory investigated compounds related to a brominated allyl phosphate polymer, a resin material reported to be effective. The search was directed toward compounds that could be prepared easily from available materials and applied as water dispersions. Two new flameproofing compounds were made that have the unique properties desired for flameproofing cotton; one is a halogenated addition product of triallyl phosphate and the other is bis-(dibromopropyl) phosphoric acid.

The two compounds were applied on a laboratory scale by methods similar to those used in commerical processing. The treated textiles possessed the excellent flameproofing qualities desired in military clothing even after a number of launderings. Investigations are being continued to improve the application of the compounds to cotton and to evaluate the processes on a pilot-plant scale.

Physical properties of chemically modified cotton fibers investigated

Improvement in the quality of cotton is of interest to the Armed Forces, to industry, and to the consumer. Alterations in the cellulose structure of fibers cause changes in their mechanical properties, which in turn affect the quality of the end product. Obviously, knowledge of the changes in properties of modified fibers can help greatly in predicting the performance of the products. To provide this information, the properties of a number of new types of chemically modified cottons, described in previous reports, were investigated.

Six commercial cottons of a wide range of physical properties were modified by acetylation, carboxymethylation, mercerization, aminization, and decrystallization. Properties such as strength and elongation were measured on individual fibers and on yarns. The properties were significantly different after chemical treament, the extent of the changes depending on the type of treatment, the tensional forces on the yarns while they were being treated, and the inherent characteristics of the untreated cotton. A physical property measured on the yarn usually differed from the property when measured on the fiber, indicating the interaction of physical properties of fibers in the yarns.

Correlation of the changes in the physical properties of the fibers

and yarns with the physical conditions employed in chemical processing and with the properties of untreated cottons provides a basis for improvement in the quality of a chemically modified textile product.

Wool

Controlled delays during the processing of wool improve spinning performance

Divergent views have been expressed in regard to the advantage of resting wool between stages in processing. Some consider aging necessary to dissipate static electricity accumulated during carding and combing. Others hold that relaxation and return of fiber crimp lost in procesing are involved. No consistent information was available that would support specific views. Such information has now been obtained, however, in investigations carried out for the Department of Agriculture by the Textile Research Institute at Princeton, N. J., under a research contract.

This research was initiated in October 1948, as a 4-year study. The major objective was to determine whether the resting of wool at various stages in processing improves the spinnability and possibly other properties of wool or wool fabrics and, if it has such beneficial effects, whether they can be obtained by means other than those requiring long periods of storage.

The project, supervised by the Western Regional Research Laboratory, included a critical examination of aging. Mill processing studies were made under carefully controlled conditions by the Forstmann Woolen Co. under a subcontract. Specialized research methods, developed at the Textile Research Institute, were employed to study the structure and mechanical properties of single fibers, yarns, and fabrics throughout processing. The study comprised comparison of an Australian wool with a domestic fine wool, and a New Zealand wool with a domestic medium wool. These wools were processed by the so-called French combing system and were studied through stages of scouring, carding, combing, drawing, spinning, weaving, dyeing, and finishing. Effects of controlled delays applied between the stages of carding, combing, and spinning were specifically considered.

These studies demonstrated that the resting of wool after carding or after drawing before spinning improves spinning efficiency. Moreover, it was found that short treatments of the freshly carded or drawn wool with steam have an effect similar to that obtained by resting the wool for long periods. The improvements produced by resting and steaming are particularly marked when yarns are spun close to the limit of fineness for the fibers involved. The effect of resting is so important under these conditions of spinning that aging for even 1 or 2 days under normal conditions of temperature and humidity improves the spinning efficiency. Improved spinning efficiency lessens the supervision required and hence leads to increased spinning economy.

MEDICINALS

Research on dextran establishes production of this important blood-plasma extender

In an extreme national emergency, it would be practically impossible to make sufficient plasma or whole blood available to meet the

heavy requirements for shock, burns, and other casualties. The potentially enormous number of casualties, especially in an atom bomb attack, makes it imperative that alternative measures be worked out as rapidly as possible. The one answer having the greatest promise of success is the development of the best possible stable plasma substitute that can be stockpiled. Only by such a procedure can we be assured that an adequate supply of this lifesaving material will be available when urgently needed.

Of the many substances studied as blood-plasma substitutes, dextran was one of the earliest and most favorably evaluated both in this country and abroad. In spite of all the research on this potentially valuable substance, information is needed on its ideal specifications and methods of analysis for control of quality, as well as information that will lead to the most satisfactory source and method of producing clinical dextran. The research summarized here is aimed at these objectives.

Natural dextrans (also called native dextrans) are produced by a number of bacteria of the family Lactobacteriacae when grown in cultures containing sucrose (cane sugar). Only the glucose part of the sucrose, however, is utilized in the formation of dextran.

Like starch, glycogen, and cellulose, dextran is a polysaccharide. It is composed of glucose sugar units linked together to form molecules of high molecular weight. In dextran, however, the mode of linkage between the glucose units differs from that in the other polysaccharides. In the molecular structure of dextran, the linkages are predominantly what a chemist refers to as the alpha-1,6-type, whereas in starch and glycogen they are the alpha-1,4-type.

Natural dextrans have extremely high molecular weights. To be compatible in the body's circulatory system after intravenous administration, a dextran must be broken down to the proper molecular weight. Natural dextran, therefore, must be partly degraded by chemical, biochemical, or physical means. The random mixture of molecules, ranging from extremely large to extremely small, must be fractionated to obtain that fraction having the proper average and range of molecular weights. To insure that such a fraction is safe for clinical use, its physical constants must be determined. This material is made into a 6-percent solution by weight in a physiological salt solution, sterilized, and bottled aseptically. After bottling, the solution must be tested biologically to be sure it can be administered without risk. The stability of dextran is such that either the dried clinical fraction or the sterile solutions can be stockpiled in large quantity.

Survey of types of dextran.—Early work (1944) at the Bureau's Northern Regional Research Laboratory on the production of dextran for investigations on the structure of starch showed that different strains of bacteria produce dextrans of unlike chemical structure. The micro-organism chosen from the laboratory's culture collection for the main studies was a vigorous culture previously isolated by the laboratory from a sample of contaminated ("ropy") root beer and classified as *Leuconostoc mesenteroides* NRRL–B–512. Dextran was obtained readily in excellent yields. It differed so much from dextrans previously described that it appeared to be a new structural type. Publication of the methods for preparing, purifying, and characterizing this particular dextran resulted in immediate interest by

many medical, industrial, and academic research institutions, both here and abroad. All domestic producers of dextran as a blood-plasma substitute are now using the NRRL–B–512 organism.

Since the start of the Northern Laboratory's large-scale program in 1950 on the production of blood-plasma expanders, an extensive survey of types of dextran available for the preparation of clinical dextran has been a phase of the dextran research. Dextrans produced by more than 100 strains of bacteria have been prepared and characterized by chemical and physical methods. The variation among these dextrans is great, and it has been shown to be due to true structural differences. Such differences are expected to affect the suitability of the dextrans for production of blood-plasma extenders, as well as for other specific uses.

Enzymatic production of dextran.—Production of natural dextran by bacteria involves two steps. The first deals with the growth of bacterial cells and the production of the dextran-synthesizing enzyme; the second involves the conversion of the remaining sucrose to dextran by the enzyme. Conventionally, these two stages occur successively in the same fermentation. An effort was made to produce dextran more efficiently by first producing the enzyme and subsequently introducing it directly into a sucrose solution void of bacterial cells. Because the enzyme exists and acts outside the bacterial cells, separating it from the cells is relatively simple.

The most serious obstacle to the use of the enzymatic process by industry has been the difficulty of procuring enzyme preparations of sufficiently high potency in adequate quantity. However, by using 2-percent sugar concentration with the NRRL–B–512 organism and by careful control of the acidity in both the development of the seed culture and the main fermentation, a suitable culture liquor is obtained. This culture liquor has a sufficiently high enzyme potency and, after it has been made slightly alkaline, can be filtered or centrifuged to remove cells and other solid material. The resulting enzyme preparation is promptly stabilized with acid to protect its potency for future use.

Small-scale studies at the Northern Laboratory indicated that for a given fermentation capacity, plant production can be doubled and greater conversion efficiency can be attained by using the enzymatic process instead of the whole-culture method. Better production controls are possible when the two phases, production of the enzyme and synthesis of the dextran, are carried on separately.

Fundamental investigations on synthesis of dextran.—Experiments showed that the molecular weight of the dextran produced by the enzyme could be controlled by the concentration of sucrose, but not well enough to obtain a good yield of a dextran having the molecular weight 60,000–75,000, which is most suitable for its use as a blood-plasma extender. Furthermore, the action of the enzyme dextransucrase is selective—it is unable to synthesize dextran from any sugar other than sucrose. Other sugars, however, especially isomaltose, maltose, glucose, alpha-methylglucoside and fructose, although they cannot be substituted for sucrose, can be used with varying results to modify the course of the conversion of sucrose by the enzyme. The sugar that had the greatest effect on the reaction—isomaltose—approximately doubled the rate of sucrose disappearance and had a significant effect on the molecular weight distribution in the product.

These fundamental investigations showed that formation of dextran can be radically altered by introducing different substances into the reaction mixture, and by changing or controlling the concentrations of the reactants. It is reasonable to assume that by these devices it may be possible to synthesize a natural clinical dextran directly instead of relying on the more costly process of degrading the dextran of higher molecular weight to obtain a fraction suitable for clinical use.

NEW DEXTRANS PRODUCED FOR CLINICAL INVESTIGATIONS.—To produce the maximum yield of a fraction of the desired molecular weight, each natural dextran requires special conditions, such as the time and temperature during the acid and thermal degradations and the concentration of alcohol used for the subsequent fractionation. Both the acid and thermal degradations of the dextran formed from the NRRL–B–512 micro-organism have been studied in detail at the Northern Laboratory.

Another problem of major importance is to find the cause of the antigenic-type reactions, which have occurred with high frequency in the clinical use of dextrans of foreign manufacture and which may be related to the structure of dextran. One approach to this problem was the development and application of suitable conditions for hydrolysis and fractionation for the preparation, in good yields, of clinical fractions from two structurally different types of natural dextrans not previously available for study. Such dextrans were successfully produced and hydrolyzed. The resulting fractions of the proper molecular weight and purity were bottled elsewhere, as a 6-percent solution in a 0.9-percent saline solution, and rendered sterile and free of pyrogens, toxins, and antigens. Clinical studies are now in progress by investigators selected by the National Research Council on Shock.

CHEMICAL STRUCTURE OF DEXTRAN.—New data developed at the Northern Laboratory showed that certain dextrans have chemical structures markedly different from those previously suspected, or reported in the technical literature. Knowledge of the fine structure of dextran molecules is essential for differentiating between types of dextran and for making correlations between the chemical structure of dextran on the one hand and colloidal properties, enzyme susceptibility, and clinical behavior, on the other.

Analysis by sodium metaperiodate oxidation has proved to be the easiest and quickest method for surveying large numbers of dextran samples and for demonstrating differences between them. More than 300 natural and partly degraded dextrans were analyzed, and their contents of alpha-1,6-linkages were found to range from 50 to 96 percent. Additional information on structure was obtained by examining fragments of the oxidized dextran after degradation by hydrolysis. These data indicated that the dextran synthesized by a second chain of *Leuconostoc mesenteroides*, NRRL–B–742, contains more than 20 percent of the unusual alpha-1,3-linkage.

Further data on the chemical structure have been obtained by methylation analysis. In the conversion of a dextran to its fully methylated ether, only the free hydroxyl groups in the molecule—those not involved in linkages to other units—enter into the reaction. When the methylated dextran is hydrolyzed, the positions of the free hydroxyl groups formed indicate points of attachment in the original

chain. In the dextran synthesized by the NRRL–B–512 micro-organism, 2,4-dimethyl glucose has been tentatively identified as a product of the hydrolyzed methylated dextran. This fact indicates not only that alpha-1,3-linkages are present but that they occur as points of branching in the dextran. Specific information on the arrangement of branches in the molecules and the length of individual branches is needed, in addition to the information regarding the kinds of glucosidic linkages present in dextran and whether they occur at branch points. This is being obtained by study of the products of partial acid hydrolysis of dextran.

This concerted research on the structure of dextran by periodate oxidation, methylation analysis, and partial hydrolysis has passed the preliminary phases, in which the development of techniques and procedures is of greatest importance; it is entering the stage in which results and correlations on the various dextrans are forthcoming. This work is important in providing a structural basis for the production of a superior blood-plasma extender.

Physical methods for characterizing dextran.—A primary requirement of a blood-plasma volume expander is that it must exert a high osmotic pressure yet remain in the circulatory system for a time sufficient to permit operation of a patient's normal recovery mechanism. High osmotic pressure at moderate concentrations requires dextran of low molecular weight, but to avoid immediate excretion of the dextran by the kidneys its molecular weight must not be reduced beyond a definite limit. These opposing requirements mean that a critical compromise must be made in the selection of an optimal molecular weight. On the basis of the limited data now available, the Army Procurement Agency has specified not only the average molecular weight for clinical dextran but also the limits for the range of molecular weights permissible in clinical material. To meet the urgent need for methods for accurately determining molecular weights and molecular weight distributions, the Northern Laboratory has developed procedures for using both osmotic pressure and light-scattering measurements for determining reliable molecular weights of dextran.

For control of the fractionation of clinical material from degraded natural dextrans, a simpler and more rapid method for determining molecular weights than the osmotic pressure and light-scattering procedures are essential. Although less sensitive, viscosity measurements on dextran solutions provide such a method if calibrated by one of the absolute methods. This relationship between viscosity and molecular weight has been established for fractions of several different acid-degraded dextrans. The viscosity-molecular weight relation developed for dextran produced by *Leuconostoc mesenteroides* NRRL–B–512 has been made available to all industrial concerns known to be working with this dextran.

Cooperation.—The Northern Laboratory's dextran research is closely coordinated with the program and recommendations of the Subcommittees on Shock and Burns and Blood and Blood Derivatives of the National Research Council; the Surgeon General's Office; the Bureau of Medicine and Surgery, Department of the Navy; Medical Research and Development Board, Department of the Army; National Institutes of Health of the United States Public Health Service; the

National Bureau of Standards; Office of Medical Services, the United States Department of Defense; and other official agencies charged with the responsibility for national health and defense.

Under a program on the animal and clinical testing of dextrans of different structural types, the Northern Laboratory has cooperated with the National Research Council by furnishing clinical dextrans prepared at the Laboratory to investigators selected by the Council. These investigators include certain research groups operating under supervised contracts and grants of the National Research Council, as well as Army hospitals, such as Walter Reed Hospital at Washington, D. C., and Brooke General Hospital at Fort Sam Houston, Tex.

Samples of natural and clinical dextrans produced by different bacterial strains have been furnished to investigators at the Cornell University Medical College for a comprehensive survey of the seriological behavior of dextrans and the possible relationship of such behavior to the "untoward" clinical reactions observed with some dextrans. For the same purpose, samples of special dextrans for immunological studies have been furnished to the Allergens Research Division of this Bureau as well as to investigators selected by the National Research Council.

Search for plant steroidal sapogenins for corsitone synthesis continued

As mentioned in the 1951 report, the Eastern Regional Research Laboratory is cooperating with other Federal agencies in a study of plant steroids as cortisone precursors. Since the last report, more than 1,800 additional plant samples have been received from the Division of Plant Exploration and Introduction, Bureau of Plant Industry, Soils, and Agricultural Engineering, making a total of more than 2,700.

Since it has been established that cortisone can be synthesized from hecogenin and diosgenin, intensive efforts have been made to find plants with a high content of sapogenins.

The best sources of hecogenin are the leaves of various species of *Agave*. Several have been found in Arizona, New Mexico, and California that contain appreciable quantities (0.3–0.8 percent), and 10 or 15 species found in Baja California and other northwestern States of Mexico contain appreciable amounts. The most promising species have been sent to the Huntington Botanic Gardens, Pasadena, Calif., for propagation and agronomic study.

The best source of diosgenin are the roots or rhizomes of *Dioscorea* (Mexican yam). Although several species that contain as much as 0.5 percent diosgenin are native to the United States, by far the best sources have been found in the State of Vera Cruz in Mexico. Because the botanical identification of the *Dioscorea* is difficult, many of the *Dioscorea* collections are as yet unidentified. There seems to be two chief types, both from regions south of Vera Cruz. One type, which resembles a large tortoise shell, is represented by *Dioscorea macrostachya* and *D. composita*. Some samples contain as much as 4 percent diosgenin, although 2 to 3 percent is more typical. A second type, unidentified, is smaller, and the rhizomes are long and thin. This type is apparently even richer, containing up to 5 percent diosgenin. Promising species are being propagated at Beltsville, Md.,

and elsewhere by the Bureau of Plant Industry, Soils, and Agricultural Engineering.

In addition to hecogenin and diosgenin, many other sapogenins were found in various species of *Agave* and *Yucca*. It was particularly interesting to discover that the byproduct leaf powder of a commercial *Yucca* fiber factory contains 1.0 percent sarsasapogenin. This sapogenin is a good source of sex hormones, and it may be a promising material for the synthesis of cortisone.

More than 1,000 samples of species of other than *Yucca*. *Agave*, and *Dioscorea* were examined, with almost completely negative results, indicating that steroidal sapogenins occur in only a small percentage of plants.

Improvements were made in the isolation and identification of steroidal sapogenins. Crude sapogenin acetates in acetic anhydride can be extracted with hexane, leaving resins and tars as a residue. This method eliminates several steps in the procedure for purifying sapogenins, in particular the benzene extraction described in the 1951 report.

All the common sapogenins have been carefully purified, and their physical constants have been determined, in many cases for the first time. In particular, the complete infrared spectra and optical rotations have been obtained for pure sapogenins and their acetates, thus greatly simplifying identification of unknown compounds.

The saponin glycoside precursors of the steroids have been isolated and partly purified. Infrared studies of the compounds and their acetates showed for the first time that the steroidal part of saponins is identical with that of the sapogenins derived by acid hydrolysis and that the particular steroids obtained are not artifacts of the hydrolysis.

Saponins vary markedly in their rate of hydrolysis by acids. Boiling 4 N hydrochloric acid completely hydrolized all saponins in 2 to 3 hours, whereas 2 N acid gave results as much as 50 percent lower. Hence 4 N acid has been adopted as the standard in this work.

It was discovered that aqueous extracts of fresh *Agave* and *Yucca* leaves and *Dioscorea* tubers have enzymes that can hydrolize the saponins in the same species. The sapogenins isolated are identical with those produced by hydrolysis with acid. The method is an improvement over hydrolysis with acid because relatively little tar is formed.

Early in this investigation it was realized that most of the plants submitted would be little-known wild plants and that an unusual opportunity would be wasted if they were not examined for nonsteroid constituents. A survey of the literature showed that there is no published chemical information on about half the species represented in the first 1,000 samples received. Another screening procedure was therefore devised by which the samples could be examined quickly for six groups of constituents—flavonols, alkaloids, tannins, sterols, organic acids, and phenols—and rated on a scale of zero to plus 3.

Among the first 1,200 samples received, a plus 3 rating was given to 84 species for sterols, 28 species for alkaloids, 19 for tannins, and 1 for organic acids. Plus 2 was assigned to 15 species for flavonols and to 1 for phenols. This information will be of considerable interest to plant chemists and pharmaceutical companies seeking new plant constituents and new sources.

INDUSTRIAL PRODUCTS

Potential new uses for animal fats evaluated in survey

To obtain information about present and projected research on fats such as lard, tallow, and grease, a survey was conducted by a consulting chemist, under a RMA contract. More than 150 companies and individuals were interviewed; patent structures of organizations working in the field were also examined.

Important conclusions from the survey are:

1. Further substantial increase in production of fatty acid will come largely from tallow and grease, the basic acids of which are palmitic, stearic, and oleic.
2. Instability in the price of tallow has discouraged research aimed at the utilization of this fat.
3. The possibility of regaining the past market for tallow by development of detergents from it is definitely limited.
4. Lard, if properly processed, is as good as a vegetable shortening.
5. Certain groups of fat derivatives promise commercial use in fields in which long-chain structure is particularly advantageous.

Epoxidized oils now produced in substantial quantities for use as plasticizers

One of the most important processes for converting fats to more useful commodities is oxidation. Accordingly, several methods for oxygenating fats were investigated at the Eastern Regional Research Laboratory, and a relatively large number of oxygenated derivatives of fats, oils, and fatty acids were prepared. As a result of these investigations, a practical, cheap method for preparing epoxidized oils has been devised that may be used satisfactorily with a wide variety of oils, both animal and vegetable. This simple method consists in the reaction of the fat with either performic or peracetic acid under controlled conditions.

Recently it was discovered that certain epoxidized oils have properties that make them especially valuable as plasticizers in vinyl plastic formulations. Particularly valuable for this purpose is the avidity of epoxidized oils for hydrogen chloride, which is usually released from polyvinyl chloride in small amounts, with resulting discoloration and deterioration of the plastic products. Consequently, epoxidized oils are now being prepared commercially for use in this field.

Tobacco stems can be made safe for fertilizers

The supply of tobacco stems has been considerably augmented in recent years by the increased consumption of cigarettes and by the decreased use of the stems for nicotine insecticides. These stems could be used directly as fertilizer if it were not for the danger of spreading disease. The residual stems from nicotine manufacture, which are sterile because of the lime and heat used in the process, are highly regarded as a fertilizer and as a conditioner in mixed fertilizers. It has been shown that infected tobacco stems and refuse used as fertilizer for tobacco land may carry tobacco mosaic virus, black shank, bacterial (Granville) wilt, and wildfire.

At present practically all treatments for inactivating tobacco mosaic virus apply to the living plant, with high moisture in the leaf and the virus in an active state. Consequently, to make the stem material safe

for fertilizer, conditions had to be determined for killing the virus in the tobacco stems. The Eastern Regional Research Laboratory, in cooperation with the Maryland Agricultural Experiment Station, has made a preliminary study of the problem, and the results indicate that sterilization of the stems should be commercially feasible.

The three factors studied were the effects of temperature, time of exposure, and moisture content of the stems on the inactivation of the virus. A fourth factor, the size of the treated pieces, probably should be considered. Leaf tobacco having about 60-percent mosaic infection was stemmed in the usual commercial manner. The broken stems ranged from 1 to 3 inches in length. Four lots were prepared, with moisture contents adjusted to 32, 24, 16, and 8 percent. These lots were again subdivided to give samples for treatment at 170°, 190°, and 212° F. for 5, 15, 30 minutes. All samples were then dried at low temperature, ground, and tested for the presence of virus by inoculating living plants of *Nicotiana glutinosa*, a species highly susceptible to tobacco mosaic. The tests were made at the Maryland station.

Because of the difficulties inherent in such studies—irregular penetration of heat in stems of various sizes, sampling errors, inoculation technique, different susceptibility of the test leaves—clean-cut results were not obtained. Certain tendencies were obvious, however, and certain conclusions could be drawn.

With 8 and 16 percent moisture, inactivation was poor, even at the highest temperature and the longest exposure. With 24 percent moisture, inactivation was poor at 170° F., but better at the higher temperatures and longest exposure. The most trustworthy set of conditions was 32 percent moisture and 212° for at least 15 minutes, and preferably 30.

Such treatment should render tobacco stems safe for use as fertilizer. The treatment would not be difficult to apply in conventional driers fitted for injection of steam.

Process for alum retannage of insole leather improved

Studies on alum retannage of insole leather were continued. Tannages conducted in a commercial tannery produced alum-retanned belly leather with no indication of cracky grain. In these tests, 200 bellies were first commercially tanned with vegetable tanning materials. Then alum retannage was applied by different procedures. Some leathers were dipped in solutions of basic aluminum acetate that contained aluminum equivalent to 2.5 to 3 percent aluminum oxide. Others were retanned by applying the aluminum acetate in solution in the tannery oiling drums in amounts needed to give 2.75 percent aluminium oxide in the finished leather. Both methods gave acceptable results.

During the finishing operations, the leathers were subjected to a prolonged "sour dip" (immersion in a fermenting solution of corn sugar and Epsom salts). This resulted in a detanning action, thus removing some of the fixed aluminum oxide. The finished leathers were lighter in color than most alum-retanned leathers, and on bending showed no tendency toward cracky grain. In these respects, there was marked improvement over some of the retanned leathers previously made in a commercial tannery. In the boiling test, however, the grain and flesh areas, which had been partly detanned by removal

of alum in the sour dip, showed considerable shrinkage, although in general the interior fiber appeared to be well retanned. To replace the aluminum oxide removed in the sour dip, the leathers were given a light dry-dip retannage at the Eastern Regional Research Laboratory with a basic aluminum acetate solution equivalent to 1 percent aluminum oxide. They were then oiled with an alum oil (an oil adapted for use on alum-tanned leather) and dried. These leathers were satisfactorily alum-retanned. The grain had greater flexibility and did not crack on bending. Analysis of the leathers showed that the aluminum oxide content varied for different pieces, depending on the thickness and character of the leather. When boiled in water for 3 minutes, the leathers with aluminum oxide contents of 1.5 percent showed moderate shrinkage; those with 2 percent showed slight shrinkage, and those with 2.50 percent showed no shrinkage.

Insoles for a shoe service test were cut from these alum-retanned belly leathers and vegetable-tanned belly leathers tanned in the same tannery. Each pair had one alum-retanned insole and one vegetable-tanned insole cut from the same location on the hide; the insoles of each pair were of uniform thickness. The insoles were built into 68 pairs of test shoes, which are being worn by cooperating postmen to determine the improved serviceability of alum-retanned insoles as compared with insoles commercially tanned with vegetable tannins.

Research on canaigre roots as a domestic source of tannin continued

Investigations were continued on canaigre as a domestic source of tannin. The agronomic phases of the studies were conducted by the Bureau of Plant Industry, Soils, and Agricultural Engineering, and investigations on processing, production of tannin extract, and tanning were made at the Eastern Regional Research Laboratory.

The canaigre field work involved chemical analyses of roots grown in the agronomic and breeding program to determine the effects of herbicides and fertilizers on tannin content, variation in tannin content of roots produced in different localities in Arizona, Texas, and New Mexico, and comparison of roots from various strains of canaigre grown in a field nursery from planting stock obtained in eight Southwestern States. Roots grown in the nursery at Queen Creek, Ariz., from California and Arizona planting stock had the highest tannin contents, ranging from 32 to 40 percent (moisture-free basis).

Studies were continued jointly in the field and at the Eastern Laboratory to develop satisfactory methods for storing freshly dug canaigre roots. Roots were stored in pits, in piles covered with earth, and in open air under sheds. In some tests, the stored roots showed a high degree of spoilage and loss of tannin. There were indications, however, that roots could be stored without substantial loss of tannin. Furthermore, the extractable tannin of some of the roots apparently increased, and the purity of the extract was improved. Further studies of this problem are under way.

Although a procedure has been developed for extracting most of the tannin from canaigre roots and preparing an acceptable tanning extract, more efficient extraction is needed. Because diffusion of tannin from canaigre roots is slow when extraction is conducted at the low temperature required to avoid gelatinization of the starch, it is important for satisfactory extraction that the roots be neither too coarse nor too fine. Wet-pulping in laboratory-scale blenders or mix-

ers that produce a shearing cut rather than a fine grind gives a preparation from which the tannin can be extracted effectively. Equipment of this type, however, is not available in capacities suitable for commercial use. A laboratory-scale beater such as that used in preparing paper pulp also gives satisfactory preparation, and since beaters of this type are available in commercial capacities, they will be studied further.

The use of yeasts to convert the carbohydrate materials—sugar and starch—of the extracted liquors into alcohol was investigated. Most yeasts tested did not survive in canaigre liquors, but eight cultures were found that could survive in 6-percent canaigre liquors.

The results of tests have suggested a method for converting canaigre carbohydrate materials into alcohol. By this method up to 95 percent of the sugar is consumed, and the yield of alcohol is about 84 percent of the theoretical value. The purity of canaigre liquors is increased from 50 to 72, which is acceptable purity for tanning extracts. It is estimated that in an extract plant that processes 183,000 pounds of fresh roots a day, about 2,000 gallons of 95-percent ethyl alcohol could be recovered.

To obtain a quick evaluation of the serviceability of sole leather tanned principally with canaigre, cattle hides were cut in half along the backbone line, trimmed to give right and left bends, and tanned. From the right bends, sole leather was produced in a tannery with a commercial blend of several tannins. The left bends were tanned at the Eastern Laboratory with a tanning blend containing 50 percent canaigre, 25 percent quebracho, and 25 percent chestnut tannin.

Garrison-type shoes were made, each pair of which had one canaigre-tanned sole and one commercially tanned sole, cut from corresponding locations on opposite sides of the same hide. Sixty-eight postmen are wearing the shoes to test the wearing quality of the leather. When the soles are worn thin, the shoes will be resoled, and in resoling, the two types of sole leather will be reversed. The results of the test should indicate any marked difference in the serviceability of the two leathers.

A semiworks plant for producing dried tannin extract from dried canaigre roots is being set up at the Eastern Laboratory to produce sufficient canaigre tannin for plant-scale tanning tests.

Extraction of the roots now available together with those to be harvested during 1952 is expected to yield 20 to 25 tons of tannin extract (12.5 to 15.5 tons of pure tannin). The data obtained from this relatively large-scale extraction should be sufficient for designing a full-scale extraction plant and estimating the cost of producing tannin from canaigre roots.

Experiments were also carried out on drying canaigre roots in alfalfa driers of a type used in some areas of the Southwest where canaigre may be grown. The roots could be dried in this drier at inlet air temperatures up to about 900° F. without material loss of tannin, provided that the temperature of the product did not exceed 212°.

Synthetic lubricants derived from soybean oil "foots"

Because of the extremes of temperature under which jet engines are used, oils for lubricating them must have pour points below −65° F. and retain their lubricating properties at temperatures as

high as 392°. At present, the base for these lubricants is a compound of sebacic acid, derived from castor oil. Because castor oil is produced primarily from a foreign crop and has critical and strategic uses, it is one of four oils being stockpiled for emergency use. If replacements for the sebacic acid compound could be produced from domestic raw materials, it would save castor oil for other critical and strategic uses.

Soybean and other vegetable oil "foots" are substantial sources of fatty acids. These acids have been used in various forms in lubricants. Although they are not satisfactory in lubricants for jet engines, these acids could be modified to give suitable replacements for the sebacic acid compound. It has been found that some of the derivatives have pour points ranging from −44° to −51° F., and they show relatively little change in viscosity. Although these properties are not superior to those of the lubricant base now used, preliminary results obtained at the Northern Regional Research Laboratory indicated that slight modifications of these compounds would produce materials that have suitable viscosity characteristics and pour points comparable with those of the sebacic acid compound. Further basic study of the molecular arrangements of these compounds should lead to modified derivatives that have the desired properties.

Azelaic acid synthesized from furfural

Because of military requirements for lubricants that function efficiently at both extremely high and extremely low temperatures, a search was made for new synthetic materials that have the desired properties. Previous research conducted by the Navy and the Air Force had shown that esters of certain dibasic acids most nearly meet those requirements. Esters of sebacic and azelaic acids have especially good properties, but these acids are derived from castor oil or other oils that might be unavailable or critically scarce in time of war. The Munitions Board was interested, therefore, in a process for producing these chemicals from readily available domestic nonstrategic raw materials. A laboratory procedure for synthesizing azelaic acid from furfural (derived from corncobs) and acetaldehyde (derived from ethyl alcohol) has now been developed by research workers at the Northern Regional Research Laboratory. This procedure is being intensively investigated to determine the best operating conditions for obtaining the highest yields at the lowest cost.

Alcohol-water injection tested in farm tractors

One of the most important potential applications of alcohol-water injection to automotive engines is in farm tractors. Large-scale tests were made with the object of determining whether unforeseen difficulties will arise when alcohol-water injection is used to boost the octane number of fuels. Its economic operation was also studied.

As shown in chassis dynamometer and field tests conducted previously by the Northern Laboratory with a special high-compression tractor engine, alcohol-water injection significantly increased the power output and economy of operation. For this high-compression tractor engine, it was necessary to use alcohol-water injection or liquid petroleum gas, because no premium gasoline had an octane number high enough for knock-free operation.

The use of alcohol-water injection in tractors was tested on farms

by the Agricultural Experiment Station of Ohio State University under a research contract administered by the Northern Regional Research Laboratory. Fifty tractors having compression ratios ranging from 5.3 to 1 to 7.9 to 1 were used in this experiment. No difficulties attributable to alcohol-water injection were encountered. Because of both the extra power and improved economy obtained, but especially the additional power, 12 of the 15 cooperating farmers using the high-compression tractor engines indicated their desire to continue with alcohol-water injection after the tests were completed. The farmers estimated that with alcohol-water injection gasoline consumption in the high-compression engines was from 15 to 30 percent less than that required by standard engines for the same work. The average consumption for year-round operation of the 50:50 alcohol-water mixture used was 4 percent of the gasoline used or 2 percent on the basis of alcohol alone.

Advances made in experimental use of wheat-straw and sugarcane-bagasse pulps for improved fine papers and newsprints

Interest in straw and bagasse pulps for blending with wood pulps and other paper-making materials has increased considerably in the United States, Europe, and South America. Partly on account of the experimental work of the Northern Regional Research Laboratory, interest in the United States has veered from all-straw or all-bagasse papers to blends of these agricultural residue pulps with the regular wood pulps used for making paper. Reports from Europe, South America, and the Orient confirm the work of this Laboratory on the suitability of these agricultural residue fibers for blending with other paper-making fibers.

Part of the increased interest in these pulps in the United States is due to the greatly accelerated consumption of paper and board products here. The large recent expansion in production of wood pulp in the South to meet this demand is causing some concern regarding conservation of our wood resources. Since practically all the expanded pulp capacity is for integrated mills, the nonintegrated mills and mills that have to go into the market for their wood-pulp supplies are looking closely at straw and bagasse as raw materials for production of paper.

In a series of cooperative paper-making experiments at the Forest Products Laboratory, pulps prepared at the Northern Laboratory from Illinois straw and Florida bagasse were blended with Eastern and Lake States wood pulps to produce magazine, book, bond, and newsprint papers. The same types of paper were made from all-wood-pulp furnishes as controls. The first group of experiments with straw pulps was discussed in the 1951 report.

In all cases, addition of the straw and bagasse pulps resulted in papers with better formation and surface and greater strength. As expected, the bulk and opacity of the papers containing straw or bagasse were slightly lower than those of the all-wood-pulp papers. Of particular interest is the fact that the use of bagasse or straw pulps in the kraft bag paper resulted in products with considerably higher bursting, tensile, and folding strength than those of the all-wood-pulp bag paper.

In newsprint papers, bleached straw and bagasse pulps were used

to replace entirely the chemical wood pulp (sulfite) normally used, keeping the ground-wood content of the papers within the normal range of 70–80 percent of the total furnish. No difficulties were encountered in producing the straw- or bagasse-containing newsprint on the Fourdrinier paper machine; in fact, the wet web had considerably greater tensile strength than the corresponding web from the all-wood-pulp furnish. The newsprint containing the straw or bagasse pulp was smoother and harder, and showed better formation than the all-wood-pulp papers. The paper containing straw or bagasse also had greater bursting and tensile strength. In all other specifications for standard newsprint, the papers were about the same.

As a result of these newsprint tests, a manufacturer with a large newsprint mill in the South became interested in blending a small amount of bagasse pulp with newsprint furnish to stiffen the normally limp paper produced from southern pine ground wood mixed with southern pine kraft pulp. After some experimental work with bagasse pulps prepared at the Northern Laboratory by the mechano-chemical process, the manufacturer arranged for a series of paper-making experiments at the Forest Products Laboratory. Experimental runs were made in which 15 to 25 percent of the bagasse pulp prepared at the Northern Laboratory was blended with 15 to 20 percent of bleached pine kraft pulp and 60 to 70 percent of pine ground-wood pulp. Control runs were also made with a blend of 25 percent bleached pine kraft pulp and 75 percent ground-wood pulp. No paper-machine difficulties were encountered in handling the pulp that contained bagasse, but there was considerable trouble in handling the wet all-wood-pulp web. The newsprint papers that contained bagasse were definitely stiffer; they closely approached standard Canadian newsprint in stiffness and in some cases surpassed it. In "blindfold" tests, the company paper experts invariably picked out the papers containing bagasse because of the stiffness and smoothness. The papers containing bagasse also had greater strength.

Encouraged by these results, the paper company is investigating the possibility of obtaining bagasse in its mill area, with the view of installing a semicommercial plant with a single mechano-chemical pulping unit for producing 10 to 15 tons of bagasse pulp a day. The use of sugarcane bagasse as a supplementary raw material for production of pulp and paper looks promising.

New processing information available for the rural grinder of corncobs, fruit pits, and nut shells

More than 24 plants in the United States are grinding corncobs commercially, and 10 are grinding fruit pits and nut shells. One plant in California processes most of the pits and shells produced in that State. Other plants are located in the Southeast, South, and Middle West.

The grinding of corncobs was a crude operation during World War II, and did not require a large investment. Today's plants may cost up to $200,000. They produce a wide variety of products, bought mostly by big industries on specifications.

Since these grinding industries are new industries, manufacturers of grinding and screening equipment had no practical knowledge of methods for processing, and consequently made mistakes in designing plants. Some manufacturers even hesitated to recommend complete

plant lay-outs. To supply the technical information required to make this new business successful, several years ago the Northern Regional Research Laboratory undertook a detailed study of the most suitable methods of grinding and classifying each of the raw materials used. It was also necessary to develop practical specifications for the products, and because of the hazards of dust explosions and fires in grinding these materials, methods of avoiding fires and explosions, as well as other safety measures, were explored.

This information was published in May 1952, in AIC–336, entitled "Dry Grinding of Agricultural Residues—A New Industrial Enterprise." The publication contains 20 tables of operating data and 18 illustrations of equipment, including a flow diagram of a grinding plant. The completeness of this work was made possible by the interest and close cooperation of operators of the grinding plants and numerous equipment manufacturers.

Improved process tested for dehydrating isopropyl alcohol

At the request of the Chemical Corps, Department of the Army, experiments were conducted at the Northern Regional Research Laboratory to develop a practical method for dehydrating 99-percent isopropyl alcohol to obtain a product containing at least 99.9 percent alcohol.

At the Northern Laboratory ethyl alcohol is dehydrated by distilling it at a pressure of 125 pounds per square inch gage, with diethyl ether as the entraining agent. The basic data obtained by the engineers employed by the Chemical Corps indicated that a similar method might be used for dehydrating isopropyl alcohol. Tests were conducted to determine optimum operating conditions. Using pressures of 100 to 135 pounds per square inch gage, the engineers obtained isopropyl alcohol with a purity of 99.98 percent. At such elevated pressure, it is possible to save on operating costs by reducing consumption of steam. The engineers concluded that the process is practical, and the data obtained in these tests will be applied in designing a large-scale distillation unit for the Chemical Corps.

High quality of guayule rubber demonstrated in heavy-duty truck tire tests

Natural rubber continues to be essential for the manufacture of heavy-duty tires for trucks and buses, since no synthetic polymer meets the critical requirements for the tire carcass. Work is therefore continuing at Salinas, Calif., on the development of new or improved processes for extracting rubber from the domestically produced guayule shrub, with the aim of producing rubber that will serve as an adequate substitute or supplement for imported Hevea (plantation) rubber in an emergency. This investigation, authorized under the Critical Materials Stock Piling Act of July 23, 1946, is conducted in cooperation with the Bureau of Plant Industry, Soils, and Agricultural Engineering.

As previously reported, research on this project has shown that the quality of guayule rubber is significantly improved, first, by processing freshly harvested shrub to avoid degradation of rubber resulting from exposure of the shrub in the field or in storage or related "conditioning," and second, by solvent extraction of the 20 percent or more of resinous material normally present in the crude rubber.

Industrial evaluation of the high-quality deresinated guayule rubber has received major emphasis because it provides the most reliable means of assessing the progress made. During the past year, approximately 4,000 pounds of the deresinated rubber was prepared by new methods for testing in heavy-duty truck and passenger-car tires to determine its serviceability in comparison with Hevea rubber. The Synthetic Rubber Division (formerly the Office of Rubber Reserve), Reconstruction Finance Corporation, made arrangements for manufacture of the tires by the Firestone Tire & Rubber Co., the Goodyear Tire & Rubber Co., and the Armstrong Rubber Co. Road tests were conducted by the Synthetic Rubber Division's test fleet at Camp Bullis, San Antonio, Tex.

The first set of truck tires, on which a complete report of tests is now available, was made by the Firestone Tire & Rubber Co. These tires were size 9.00 x 20, 10-ply rating with rayon fabric. Carcasses of three compositions were compared with a 100-percent Hevea carcass standard. They included a blend of 45 parts each of Hevea and guayule with 55 parts of GR–S synthetic rubber, in addition to the 100-percent guayule carcass. Three tires of each type were tested. The treads in all cases were fabricated of "cold" GR–S rubber, and the tubes were butyl rubber. The tires were mounted on the rear wheels of similar model trucks, loaded with gravel ballast to 145 percent of the maximum recommended for such tires. Running speeds were 45 miles per hour on pavement and 30 miles per hour on gravel.

According to the final report issued by the Synthetic Rubber Division, guayule rubber was regarded as equal to Hevea in tire-building characteristics. Road tests showed that the temperatures of the running tires were essentially the same for guayule and Hevea, indicating equivalence with regard to build-up of heat. The 100-percent guayule and 100-percent Hevea carcasses were equal in durability, as judged by one tire of each that had run 50,900 miles when the test was discontinued. Records of average miles to failure of the other two tires of 100-percent guayule or Hevea showed that guayule is equal to or better than Hevea in durability. The guayule and Hevea blends with synthetic rubber were also equivalent in serviceability, although considerably inferior to the respective 100-percent natural (Hevea or guayule) rubber tires. The cold rubber treads were approximately equal in resistance to cracking on both guayule and Hevea carcasses, with a slight advantage in favor of guayule.

Handling and utilization of tung aided by research

PROMPT DRYING OF HULLED TUNG FRUIT REDUCES FORMATION OF FREE FATTY ACIDS.—Research at the United States Tung Oil Laboratory, Bogalusa, La., yielded valuable information on the formation of free fatty acids in stored tung kernels and seeds. Knowledge regarding free fatty acids in the oils of stored tung fruit is needed by tung growers and processors to assist them in the production of high-grade tung oil; current specifications for tung oil limit the free fatty acid content to 4 percent. The trend toward hulling in the field is increasing, but tung farmers lack adequate drying and storage facilities, as well as information for safe handling of hulled tung fruit on the farm.

When unbroken kernels containing 12 percent moisture and whole

seeds of equivalent moisture were stored for 4 weeks at 100° F., the free fatty acids of the oils extracted from them did not exceed 2 percent. When similar kernels and seeds were stored for the same period at room temperature. the free fatty acids of the oils did not exceed 0.5 percent. After broken kernels of equivalent moisture content were stored for only 4 days, however, the free fatty acid content of the oil from the material stored at 100° was 4 percent and that from the material stored at room temperature was 2.6 percent. At lower moisture contents, the amount of free fatty acids was less in all three materials.

These investigations show that it is not advisable to store moist material hulled from wet tung fruit on the farm. Hulling in the field should be correlated with mill operations, so that moist hulled fruit can be taken at once to mills having drying equipment for reducing the moisture content. Because of the tendency for free fatty acids to develop in broken kernels, special care must be exercised in hulling to avoid excessive breakage.

Technique perfected for characterizing and evaluating tung oil.—Scientists at the Southern Regional Research Laboratory devised a method for measuring accurately the total unsaturation of tung oil by its reaction with hydrogen. Values for unsaturation obtained by this method are expressed on the same basis as iodine values to permit direct comparison with values obtained by the well-known halogen addition methods. The unsaturation in fats and oils is customarily determined by these methods, but the experimental value for tung oil is only approximately two-thirds of the actual value, because the halogen addition methods are incapable of measuring all the unsaturation.

The method determines the amount of hydrogen that will unite chemically with a given weight of tung oil in the presence of a catalyst and a solvent. It affords a means of obtaining accurate data essential for calculating the fatty acid composition. The method should be equally applicable for the same purpose to plant or animal lipids that have the conjugated unsaturation characteristics of tung oil, and should aid research leading to more efficient utilization of these materials.

World scientific literature on tung surveyed.—A bibliography comprising abstracts of about 3,000 articles and patents relating to the tung tree, the processing of the fruit, and the chemistry and technology of the oil and meal was compiled by the Southern Laboratory and the New Orleans branch of the United States Department of Agriculture Library. Published as AIC–317, entitled "Abstract Bibliography of the Chemistry and Technology of Tung Products, 1875–1950," it comprises 4 volumes, and includes more than 800 pages. Volumes I, II, and III contain abstracts of more than 2,200 technical publications and 700 patents, obtained by a review of world-wide technical literature. Volume IV consists of author, subject, and numerical patent indexes. This bibliography is the only up-to-date and comprehensive survey in English on the subject. It will be of special value to research workers dealing with drying oils and to the coatings industry.

Hard, carnauba-like wax obtained from rice bran

Scientists of the Southern Regional Research Laboratory have obtained a hard, carnauba-like wax from crude rice-bran oil. Al-

though not quite so high melting or so hard as typical carnauba—the hardest wax in commercial use—this potential byproduct of the rice-milling industry has physical properties that should give it commercial value as a hard vegetable wax.

When crude rice-bran oil, which usually contains from 1 to 2 percent of hard wax, is allowed to stand, the suspended and dissolved wax separates in the form of a semiplastic sludge. In addition to wax, this sludge contains considerable oil, particles of rice bran, trash, gums, resins, degraded organic matter, and appreciable quantities of phosphatides.

Two procedures were developed for processing the tank settlings or sludge to obtain a hard, high-melting, nontacky wax. One consists in removing the oil from the sludge by adding acetone and filtering off the acetone-insoluble material (which includes the wax), decomposing the phosphatides in the acetone insolubles by hydrolysis or saponification, and finally separating the purified wax from a solution of the crude material in isopropyl alcohol. The other procedure involves heating the sludge to 160° F., adding a small proportion of water to precipitate the phosphatides, removing the water and phosphatides, and finally separating the purified wax from a solution of the oil portion in isopropyl alcohol. Yields of hard wax from a typical sample of tank settlings ranged from 8.3 to 13.7 percent, based on the weight of the original settlings. The lowest melting point was 75.3° C.; the highest was 79.9°. Practically white waxes were produced readily by bleaching with hydrogen peroxide or with chromium trioxide in the presence of sulfuric acid solution or with combinations of these reagents.

More than 16 million pounds of carnauba wax was imported into this country in 1951, at a cost of more than a dollar a pound. With crude rice-bran oil being produced at the rate of more than 7 million pounds a year, it is reasonable to assume that a wax byproduct of high quality would be a welcome addition to the usable products derived from the rice crop. The rice-bran oil recovered from the 1951 crop would have yielded 100,000 pounds of wax. If all the rice bran of the 1951 crop had been extracted, it would have furnished an estimated 50 million pounds of oil, from which almost 1 million pounds of wax could have been recovered.

Premium-quality plasticizers made from aconitic acid, a byproduct of sugarcane

A process for recovering aconitic acid in the form of its calcium-magnesium salt from sugarcane molasses, developed at the Southern Regional Research Laboratory several years ago, has been described in previous reports. In use commercially since 1946, the process has made possible the production of this valuable byproduct on a steadily increasing scale. Large quantities of the aconitate have been produced during the nongrinding season by processing blackstrap molasses received from other factories.

The salt of aconitic acid, produced at the rate of more than 1 million pounds a year, is in demand by chemical companies for making esters used as plasticizers ("softeners") for vinyl-type resins, and for the manufacture of wetting agents or detergents. Relatively few of the esters of aconitic acid had been prepared, however, and some of them

had not been produced in sufficiently high purity for use as plasticizers of premium grade. To increase the utilization of this byproduct, the Southern Laboratory has investigated many esters of aconitic acid. These esters were transformed by a simple process—hydrogenation—into the corresponding esters of tricarballylic acid. It was found that crude aconitic acid esters can be efficiently hydrogenated without distillation or special purification to produce triesters of tricarballylic acid.

Relatively inexpensive commercial alcohols were used in making the esters. Esterification methods were improved to obtain complete reaction and high yields of the triesters of aconitic acid.

Equipment was designed that distills esters of both acids at extremely low pressures, yet effectively separates pure products without decomposition. In this equipment, it is possible to distill extremely high boiling products such as trioctyl aconitate and trioctyl tricarballylate. The high-boiling esters are most desirable because of their permanence in the finished resins and plastics.

The esters of both aconitic acid and tricarballylic acid were evaluated as plasticizers. Plasticizers or softening oils are essential for the production of resin, plastic, and certain rubber articles; they impart softness, flexibility, toughness, and other special properties to the finished products. About 225,000,000 pounds of plasticizers are used annually, and it is expected that the need for plasticizers for both civilian and military products will increase greatly during the next few years.

The evaluation studies indicated that all the esters are equal to or better than dioctyl phthalate, a widely used commercial plasticizer. Some properties of the hexyl and octyl esters appeared to be markedly superior to those of the commercial plasticizer. The tricarballylic acid esters were somewhat superior to those of aconitic acid. The preferred tricarballylic acid esters, which can be produced by hydrogenation at a slightly increased cost, are more stable in distillation and obtainable in higher purity. The data obtained in these studies have been made available to a large number of companies that manufacture resins, plastics, or rubber products. Numerous inquiries have been received for further information, and samples of the products have been requested for more thorough testing. A manufacturer of rubber goods thoroughly evaluated eight of these products, and selected three of them for further testing in a specialized application in the rubber field.

Esters of both aconitic and tricarballylic acids were supplied to research laboratories of the Armed Forces for determination of their properties as synthetic lubricants. Results of these tests led to the synthesis of esters with modified properties that might meet the critical requirements for such lubricants in certain military equipment.

A survey showed that the entire Louisiana sugarcane crop contained approximately 17,000,000 pounds of aconitic acid. Of this amount, at least 6,000,000 pounds could be recovered by the current simple process, by the addition of relatively small and inexpensive recovery units at strategic factories in the Louisiana sugarcane-producing area. With expanding production, the cost of aconitic acid from this natural source could be reduced.

New metal resinates made from gum rosin studied for specific uses

Rosins of all types are produced at the rate of more than 1 billion pounds a year, and one of the most important outlets for this commodity is in the form of metal resinates. Improved metal resinates, prepared by the Naval Stores Research Division for the varnish industry, were described in the 1950 report. The improved products are obtained by modifying gum rosin with an aldehyde, then fusing this rosin derivative with a suitable metal compound. The new resinates have higher metal contents, greater solubility, and greater resistance to oxidation than commercial resinates. They have been well received by the industry.

Bureau investigators have continued research to determine the best conditions for producing the improved resinates for specific markets. The use of a particular resinate depends not only on the metal compound used and on the amount combined with the rosin but also on the conditions for carrying out the chemical reaction to obtain desired physical properties. For surface coating and varnish resins, color and melting point are the important characteristics; for fungicides and catalysts, the metal content is more important. Good solubility in hydrocarbon solvents is always desirable. Calcium and zinc produce pale resinates with high melting points, desired for printing inks and surface coatings, including varnishes. Cobalt, manganese, and lead are used in resinates to be employed as catalysts for drying unsaturated vegetable oils in paints; and calcium, lithium, and aluminum are used in resinates for greases. Copper resinates are preferred as fungicides and as mildewproofing agents.

Because of their high metal content and good solubility, the metal naphthenates have recently replaced commercial metal resinates for some uses. Improvement of these qualities in the new resinates may enable rosin to regain some of the lost markets. The modified products would be adequate replacements if the naphthenates should become scarce in a national emergency. Several of the new resinates are being evaluated by the Forest Service as wood preservatives. Zinc and calcium resinates are being evaluated by the Naval Research Laboratory as corrosion inhibitors. Before the new metal resinates can be fully utilized, however, much work must be done on the chemistry of the reactions involved, on the development of the process for commercial use, and on the evaluation of the new products.

Synthetic lubricants for military and industrial uses developed from gum turpentine

Synthetic lubricants for military and industrial uses can now be made from turpentine as a result of 2 years of cooperative work by the Bureau's Naval Stores Research Division and the Naval Research Laboratory of the Department of the Navy.

The Navy's Bureau of Aeronautics sponsors a long-range, basic research program on synthetic lubricants, which is being carried out by its Office of Naval Research. As a part of this program, the Naval Research Laboratory is seeking sources of lubricants for aircraft gas-turbine engines, and also for use as instrument oils and greases and as hydraulic lubricating fluids, to replace sebacic acid, a dibasic acid made from castor oil, which is largely imported.

To develop domestic sources of dibasic acid for the Navy, scientists

of the Naval Stores Research Division and the Naval Research Laboratory began cooperative work on esters of acids obtained from pine gum. Esters of both monobasic and dibasic acids were tested. The esters of pinic acid—a dibasic acid made from alpha-pinene by a two-stage oxidation procedure—have the properties needed in lubricants for turbojets and in special plasticizers. In contrast with the conventional petroleum lubricants, the new synthetic lubricant remains liquid at temperatures as low as −75° and is stable at high temperatures.

These desirable properties of pinic acid and its esters are promising also for such nonmilitary uses as plasticizers, alkyd resins, and polyamide fibers. The present laboratory method of making pinic acid consists in successive oxidations of alpha-pinene by potassium permanganate and bleaching powder. Although fairly simple, this method is not economically feasible for commercial use. Further work is being done to obtain better yields and to develop methods that will produce it at lower cost. When a low-cost process is available, extensive utilization of pinic acid seems assured. Several companies have expressed interest in producing it.

Commercial production of pinic acid from turpentine is desirable because turpentine is a domestic raw material available in large supply. Besides supplying a replacement for an essential lubricant, production of this acid would help the naval stores industry by providing an outlet for turpentine. The demand for dibasic acids as sources of synthetic lubricants and hydraulic fluids is expected to require millions of pounds annually. The present consumption of dibasic acids, for all uses, is estimated at more than 300 million pounds a year.

BASIC PIONEERING INVESTIGATIONS

Cereal and Forage Crops

Tests for milling quality promise aid to wheat breeders and flour millers

Methods for determining milling and baking properties of wheats at an early stage in breeding experiments, when only small samples are available, have been needed for many years. When new strains of diseases become prevalent, and rapid development of resistant varieties is required, the need is urgent. Breeders, farmers, and millers are anxious that the new, agronomically good varieties shall be of high milling quality. When millers reject or discount such varieties, the farmer who grows them takes a loss. The 1951 report described this problem and the coordinated effort to identify the factors responsible for differences in milling behavior undertaken by the Western Wheat Quality Laboratory of the Bureau of Plant Industry, Soils, and Agricultural Engineering at Pullman, Wash., and the Western and Northern Laboratories of this Bureau.

Studies of seven Pacific Northwest wheat varieties, ranging from excellent to poor in milling quality, are in progress at the Northern Laboratory. It has been found that varieties differ in the thickness of the cell walls of the endosperm, the part of the wheat kernel from which flour is made, and that the thinner the cell walls the better is

the milling quality of the variety. This is the first indication that it may be possible to predict milling quality early in the breeding program, when only a few kernels of wheat are available for study. Agricultural experiment station wheat breeders in the Pacific Northwest are so eager for the information that they are already surveying their available material by this method, even before its applicability to more than the seven varieties studied has been determined.

Because microscopic measurements of the thickness of endosperm cell walls are laborious and time consuming, microscopic chemical tests were also investigated at the Northern Laboratory. The cell contents were removed from thin sections of wheat kernels, leaving only the cell walls. The walls were then treated with dilute acid or alkali to remove most of the water-insoluble hemicelluloses, which constitute about 25 to 55 percent of the total cell wall. The size of the area of the cell walls degraded or broken down by either the acid or the alkali was roughly correlated with the milling quality of the wheat variety. Varieties of excellent milling quality showed large areas of degradation; those of poor milling quality showed only small areas. The treatments, however, were not sufficiently sensitive to distinguish wheat varieties of intermediate milling quality. An attempt is being made to improve the sensitivity of the tests. It was shown that the chemical composition of the water-insoluble hemicelluloses of the endosperm cell walls is not correlated with milling quality.

In further studies on these soft wheats, the Western Regional Research Laboratory developed a chemical procedure for estimating milling quality that appears worthy of extensive trial. Ground wheat was extracted by a standard mild acid treatment, and the amount of pentosan in the acid extract was determined. Wheats of good milling quality contained less soluble pentosan than those of poor quality. When this method was applied to 39 samples, representing 10 varieties grown at 4 stations, the results correlated well with milling behavior. In marked contrast, other factors (weight per bushel, protein content, crude fiber content) showed no relation to the milling properties. The method requires only small samples; a 100-milligram sample of whole wheat, equivalent to 3 or 4 kernels, was used in these studies. As yet, the test has not been used with other types of wheat.

Several observations led to the development of this test. The properties of endosperm rather than those of bran appeared to be of major importance to milling behavior. Flour millers had reported that the cell-wall material of the endosperm is concentrated into a white or "fuzzy" shorts fraction that causes difficulty in milling, and such material characteristically has a high content of pentosan. Also, in earlier work on baking properties, pentosans separated from flour had been found difficult to grind.

In working with whole wheats, however, the effect of bran coats, which contain more pentosan than does endosperm, had to be overcome. Comparison of acid treatments of different severity indicated that mild acid extraction would be successful, because mild extraction removes a higher percentage of endosperm pentosan than bran pentosan. Examination of mill products (flour, shorts, and bran) from wheats that differed in milling behavior supported these results. With mild extraction, which removed amounts of pentosan reflecting milling behavior from whole wheats, the bran samples contributed

less than half the total pentosans extracted from all three fractions of each wheat. With more severe extraction, which did not extract amounts reflecting milling behavior, six-tenths or more of the total pentosans extracted was contributed by the bran samples. Furthermore, mild extraction removed a lower percentage of pentosan from each bran sample than from the corresponding sample of shorts, whereas under more severe conditions, the reverse was true. Because the shorts contain that portion of endosperm most resistant to grinding to the fineness of flour, these results also are in accord with the indications of the major importance of endosperm properties.

The procedure used in obtaining these results does not distinguish between the pentosan from the endosperm cell wall and that from other parts of the endosperm. Results obtained at the Northern Laboratory showed clearly that the variation in the thickness of endosperm cell walls is inversely related to milling quality, and they also demonstrated the large proportion of pentosan in these cell walls. The observations in the two laboratories are in accord in all respects and are definitely complementary. Both wheat breeders and millers have been outspoken in their appreciation of these researches and their results. The possibility of culling out poor-milling varieties early in the breeding program is attractive. Not only would it leave more time and money available to devote to promising material but it would also prevent the release of poor-milling varieties to farmers.

Basic information sought to retard bread staling

A major problem of the Quartermaster Corps, Department of the Army, is to prevent canned bread from becoming stale. At the request of the Quartermaster Corps and under contract with that agency, the Northern Regional Research Laboratory investigated changes that take place during staling as a foundation for developing methods of preventing or retarding the staling processes. Although staling includes both the change in flavor and the change in texture that occur as bread ages, only the change in texture, particularly the increase in firmness, was studied. It was discovered that crystallization in the starch and transfer of water from gluten to starch lead to firming of bread.

To determine the effect of aging on the arrangements of the molecules, mixtures of starch and water, which comprise about 90 percent of the bread loaf, were examined by X-rays. Starch-water mixtures that contained water in amounts customary for bread were heated under conditions simulating baking, and stored in sealed containers for periods up to 2 weeks before examination. It was found that the heating process largely destroys the natural crystallinity of starch, but in a week the crystallinity returns spontaneously. This redeveloped crystallinity may take one of three structural forms, depending on the moisture content at the beginning of storage. The crystallization processes undoubtedly cause the starch to harden and are basic to the increase in firmness of bread the first week after baking and the decreased wettability of stale bread.

Microscopic studies of canned and commercial bread showed further that the continuous framework of bread is composed of gluten; the starch is imbedded in this matrix. Changes in the gluten, therefore, as well as in the starch, must be involved in the pronounced in-

crease in firmness of stale bread. Gluten-starch-water and starch-water mixtures were prepared, heated to simulate baking, then stored in sealed containers. By conventional procedures for testing the staleness of bread, modified for use in measuring changes in gluten-starch-water and starch-water systems, it was found that aging effects are more pronounced in the presence of gluten.

Study of the effect of the simulated baking treatment on the water-absorbing properties of gluten and starch showed that it does not affect gluten, whereas it causes starch to absorb more water. Hydration of the starch, with consequent dehydration of the gluten, would be expected when gluten-starch-water mixtures that contain the same proportion of water used in bread are heated. This drying of the gluten would increase the rigidity of the gluten framework, and this process might well account for the rapid increase in stiffness of bread immediately after baking.

Knowledge of alfalfa composition expanded

Forage crops furnish approximately half the total livestock feed used in the United States. About two-thirds is consumed as pasturage, one-third as harvested hay. Total production of hay in recent years has amounted to 100 million tons annually (cured weight), including 40 million tons of alfalfa. Alfalfa is the preeminent forage crop because of its high nutritional value and beneficial role in soil conservation and crop rotation. Present methods of field-curing and handling alfalfa result in a loss of nutrients estimated at 20 to 25 percent. As a basis for methods of reducing this loss, equivalent to 8 to 10 million tons of cured alfalfa, more knowledge is needed of the composition of the plant and the changes its components undergo during processing and storage under various conditions.

About 75 percent of the dry weight of alfalfa is composed of constituents such as fats, proteins, ash, fiber, starch, and sugars; the remaining 25 percent is a complex mixture about which little is known. One-half of this fraction is water-soluble, and the presence of such constituents as vitamins, flavonoids, alkaloids, saponins, tannins, and other physiologically active materials has been either established or indicated by analytical studies. Feeding experiments and chromatographic examination at the Western Regional Research Laboratory showed the presence of at least 12 constituents, 3 of which are presumably flavonoids. Two of the flavonoids were obtained in crystalline form. One is similar to or identical with tricin, a compound isolated by British and Canadian workers, and suggested by them as a possible causative factor in bloat of cattle.

Saponin, another important water-soluble component of alfalfa, has been suggested as responsible for the inhibition of growth caused by some alfalfas when fed to chicks in amounts much above 5 percent of the diet. Investigations at the Western Laboratory revealed that the saponin content of alfalfa is 0.5 to 1.0 percent of the dry plant material. It can be isolated in a relatively pure form by the formation of an addition compound with cholesterol, followed by decomposition of this compound. Hydrolysis of the saponin showed that the molecule contains three sugars—glucose, arabinose, and xylose. Extended hydrolysis of the saponin produced crystalline sapogenin, but it has not been characterized.

Assays of a water extract showed that alfalfa is as rich as yeast in an unknown nutritional factor that stimulates the growth of *Butyribacterium rhettgeri.*

The lipid fraction of alfalfa contains carotene, chlorophylls, fats, waxes, and incompletely characterized substances. Two of the latter are the xanthophylls and antioxidants. Study of this fraction showed that it contains at least two antioxidants in addition to the tocopherols. These two constitute more than half the antioxidant content of alfalfa. Concentrates of eight hundred-fold have been prepared.

Although these studies on composition are only in the preliminary stage, the information thus far obtained and the promising leads will be valuable in the development of better methods of processing alfalfa and utilizing it as a feedstuff. Many of the techniques developed in this study will be applicable to the study of other forages.

Cotton and Other Fiber Crops

Advances made in fundamental knowledge of cotton fiber

In studies to improve cotton or to find ways of using it more advantageously, new fundamental information is being continually accumulated. Although such information does not normally have an immediate practical application, it contributes to an understanding of the properties of the fiber, and helps to predict the possible effects of processing treatments. Additional valuable knowledge was gained this year in research on cotton fibers at the Southern Regional Research Laboratory.

Nature of the primary wall of cotton fiber.—The physical and chemical nature of the outer protective membrane of the cotton fiber, called the primary wall, was studied by microscopic methods. As the first step, a technique was developed and perfected for isolating the primary wall in sufficient quantities for microchemical analysis. Investigations with the electron microscope showed that the purified primary wall consists of a loose network of fine cellulose fibrils with diameters of 200 to 400 angstroms. This structure contrasts with the strongly parallel orientation of the tightly packed cellulose fibrils that make up most of the fiber. Chemical analyses of the primary wall showed that it was about 54 percent cellulose and that the noncellulosic constituents consisted of about 14 percent nitrogenous matter (expressed as protein), 9 percent pectic material, 8 percent alcohol-soluble materials, 4 percent cutinous substances, and 3 percent ash.

Surface-adsorption properties of cotton fibers.—The surface-adsorption properties of cotton fibers are important in commercial processes employed in dyeing, chemically modifying, and impregnating fabrics to impart such qualities as resistance to water, weathering, and microbiological attack. When cotton is wet with water, it swells; on drying, it returns to its original state. This deswelling can be prevented, and the enlarged surface areas can be retained to a great extent by removing the water by solvent exchange, in which the water is displaced with methanol and the methanol with pentane before the fibers are dried under anhydrous conditions.

The National Bureau of Standards, under a contract supervised by the Southern Laboratory for the Department of Agriculture, deter-

mined the surface areas of water-swollen cottons and of modified cottons dehydrated in this manner. The areas ranged from four to several hundred times those of the original, dry cotton, depending on the sample and its pretreatment. For example, mercerized cotton showed a fourfold increase over untreated cotton in the surface available after swelling. These values are an approximate measure of the surface made accessible by swelling. They indicate, therefore, the relative dye-absorption capacities, chemical reactivities, and penetrabilities of various cottons or modified cottons in water, and similar characteristics. In addition to their practical aspects, these results contribute to a better understanding of the submicroscopic behavior of cotton.

Molecular weight of cellulose.—Although great advances have been made in recent years in knowledge of the cellulose molecule, much remains to be discovered. The increase in knowledge has gone hand in hand with new methods. It is generally accepted that cellulose consists of long, unbranched chains made up of cellobiose units, each of which contains two disklike anhydroglucose members. It is believed that the chains are relatively stiff, regularly kinked, and when sufficiently long, in solution curl up into a loose ball. In revised estimates made possible by improved methods, the value accepted for the length of cellulose molecules in their native state has been greatly increased. Work at the Southern Laboratory indicated that the cellulose molecule may be even much longer than previously reported. To learn more about the subject, attention was given to improvement of methods for determining molecular weight. Two improved viscosity techniques were developed and have been adopted by the American Society for Testing Materials.

Viscosity techniques are not capable of furnishing absolute measures of molecular weight, but must be calibrated in terms of other absolute but less convenient methods. Special studies were undertaken with the aid of an ultracentrifuge to calibrate more accurately the new viscosity techniques for investigating extra-long cellulose molecules. Preliminary results indicated that the average cellulose molecule in cotton that has not been degraded contains more than 5,000 glucose units per chain; if stretched out, it would be about 2.5 microns long and, were it not for its exceeding fineness, could be seen under the microscope. Whereas 1 mole of water weighs about two-thirds of an ounce and occupies about one twenty-fifth of a pint, 1 mole of cotton cellulose weighs nine-tenths of a ton and would occupy nearly 20 cubic feet.

Electrical properties of wool at microwave frequencies investigated

It has recently been found at the Western Regional Research Laboratory that apparatus and methods developed for use in radar can be used to investigate the fundamental electrical properties of wool, such as the dielectric constant and dielectric loss in the microwave region. The microwave band of frequencies lies between the upper end of the radio-frequency band and the lower end of the infrared band. Study of the electrical behavior of fibers such as wool and silk in this previously uninvestigated portion of the spectrum yielded new clues concerning the structure of wool. Until recently dielectric measurements at microwave frequencies could not be made because sources of microwave radiation were not available. For the work on fibers, modified

forms of radar equipment were used. In addition, a special apparatus, called a cavity resonator, was developed for measuring the electrical properties of wool fibers at microwave frequencies. This apparatus consisted essentially of a slotted cavity resonator and a frame to support a bundle of wool fibers.

The microwave region is of special interest for fibers such as wool, for which the dielectric constant is large at the highest radio frequencies. Wool apparently contains a polar unit small enough to follow the rapid alternations of radio-frequency fields. To characterize or identify the part of the wool molecule responsible for this behavior, electrical measurements must be made at frequencies in the range in which the polarizable group fails to follow the alternations of the electric field.

It was found that at a microwave frequency of 3,000 megacycles per second, the polarizable group is able to follow the alternations of the electric field almost as well as it does at one one-thousandth of this frequency. This finding indicates that the high value of the dielectric constant in the microwave region is not caused by an ordinary dipole; it may be due to the hydrogen bonds that hold adjacent protein chains together.

The new equipment is being used also to investigate the molecular interaction between water and wool. Because small amounts of absorbed water markedly affect the physical properties of wool, it is important to know the details of the sorption process. Moreover, a number of instruments employed in the textile industry use dielectric properties as a measure of the moisture content of fibers, yarns, and fabrics. These studies showed that at microwave frequencies the dielectric constant of water attached to polar groups in the wool undergoes a twentyfold change. Consequently, at these frequencies the dielectric constant is a sensitive index to the interaction between wool and water molecules. In addition it was found that the polarizability of wool is independent of moisture content between zero and 16 percent. These findings help to clarify important details of the molecular interaction between wool and water and also provide data needed for the design of an improved electric moisture meter.

Fruits and Vegetables

Further information on nonenzymatic browning of foods

Fundamental studies at the Northern Regional Research Laboratory on the chemical reactions of dextrose sugar (corn sugar) with protein-like compounds led to the discovery of new reactions and new compounds. With this information as a guide, a new theory has been developed to explain the interaction of sugars and proteins in foods and feeds that may result in serious losses in feeding value. The usual evidence of this type of deterioration is a brown color—browning—which increases with time.

The processing, storage, and conservation of feeding value of concentrated foods and feeds is of practical importance, especially when these products must be stored in warm climates or for long periods. One type of browning of sugars and proteins is entirely a chemical reaction; it should be distinguished from another type, in which the brown color is produced by enzymes. The latter type occurs in freshly cut potatoes and fruits. The browning of peaches is an example.

In chemical browning, the interaction of the proteins with the sugars leads to decomposition of the essential amino acids of the protein, thus causing a loss of nutrients in the food or feed. Although chemical browning results in loss of protein, at times a certain amount of browning is desirable. An example is the brown color and pleasant odor of fresh bread crust, which makes the bread more acceptable. Only a small degree of sugar-protein reaction is required to impart favorable odors, flavors, and colors to many food products.

Recent research at the Northern Regional Research Laboratory has shown that the first product of sugar-amine interaction (an N-glycoside) spontaneously undergoes a molecular rearrangement in the sugar, called the Amadori rearrangement, after the name of its discoverer. The rearrangement product (1-desoxy-1-amino-2-ketose) is then decomposed, forming products such as aldehydes, unsaturated sugar derivatives, and sugar degradation products of small molecular weight. All these in turn react with proteins or amino acids and with themselves to produce complex end products having the typical brown color. The addition of sulfur dioxide delays the formation of the brown color but does not completely stop the reaction.

At the same time, the primary rearrangement product degrades amino acids, forming carbon dioxide and aldehydes. Also, the primary rearrangement product, by loss of water, gives rise to reactive chemically reducing compounds known as reductones. This part of the reaction may produce substances useful as antioxidants for food preservation.

On the basis of these actions, a theory has been formulated that includes most of the previously existing hypotheses for chemical browning. This theory is expected to provide the starting point for numerous new researches, both for the detailed explanation and the control or prevention of this reaction in concentrated foods that contain both carbohydrates and proteins.

New facts about vital enzymes in plants

What is called life in biological tissue is apparently the total result of many enzymic reactions in integrated systems. Enzymic reactions continue in agricultural products after the products are harvested. But these reactions get out of balance because the substances on which enzymes act and enzyme-regulating substances are limited in the harvested tissues. Consequently, changes take place in the tissues. Some, such as the ripening of an avocado, are desirable; others, such as the softening of an apple, are undesirable.

At present the application of enzymology to the changes that agricultural commodities undergo in storage or processing is necessarily empirical, because there is so little knowledge of enzyme activity. The first phase of a systematic investigation of enzymes in plants has been started by the Western Regional Research Laboratory, in cooperation with the California Institute of Technology.

A study was made of the steps by which starch and glucose in plants are broken down and oxidized. These steps have been known for nonplant tissue, but application of this knowledge to plants had not been wholly acceptable because the existence of certain enzymes theoretically required in the scheme had not been demonstrated. The collaborative study has resulted in the discovery of the four enzymes required—hexokinase, phosphofructokinase, myokinase, and phospho-

glyceric acid kinase—and establishment of the classical Emden-Meyerhof scheme for plants.

To identify the various products resulting from each step, a paper chromatographic procedure was developed for use with radio isotopes. By this technique, it was shown that when plants oxidize certain plant acids (as well as one of the degradation products of glucose, pyruvic acid), the energy produced is stored in a chemically useful form as adenosine triphosphate, as in most other living cells. The chromatographic technique also revealed some unexplained glucose degradation products, suggesting an alternative scheme of carbohydrate utilization.

Enzymes are proteins, and a portion of the protein of each cell is made up of the many enzymes needed to maintain life in the cell. It was found that during storage plant tissues such as leaves lose a substantial amount of their protein but apparently not their enzymes. This finding may explain why the deteriorative processes to which agricultural products are subject after harvest are not sharply self limiting.

Starch of wrinkled-seeded peas contains unusual type of amylopectin

The wrinkled-seeded or common garden pea contains about 35 percent of starch that has a high content of amylose (70 percent), the film-forming fraction of starch. Since this type of starch holds promise for use in sizing and food films, basic studies on it were undertaken.

Starch low in nitrogen was prepared by the usual wet-milling process, followed by resuspension in dilute alkali. The starch was dispersed in a water medium, and the amylose was precipitated with amyl and butyl alcohols. Amylopectin was recovered from the remaining solution.

The molecular weight, viscosity, and iodine adsorption of the two fractions from the starch were determined. The properties of the amylose were similar to those of amyloses from other starches. On the other hand, the molecular weight of the amylopectin was less than a tenth of that reported for amylopectins from other starches, or about 150,000 as compared with 2 to 3 million. The individual branches making up the structure of the amylopectin averaged 36 glucose units instead of the average of 26 to 30 units in most other amylopectins.

The significance of these findings is indicated by the strength of the films of the starch acetate. Starch acetate from wrinkled peas formed films only 9 percent weaker and slightly more flexible than films prepared with pure amylose acetate. Films prepared with starch acetate from smooth peas and corn were at least 30 percent weaker and also more brittle than films prepared with pure amylose acetate. These results show that the whole starch from wrinkled peas is potentially useful for certain types of films for food and industrial purposes. It may be possible, therefore, to eliminate the expensive fractionation procedure required for other starches.

A new test was developed to measure the purity of each of the fractions. Filter paper is treated with stearic acid and spotted with solutions of starch or starch fraction; then an iodine-potassium iodide solution is added. Amylose forms a small dark-blue spot, whereas amylopectin spreads out in a large brownish circle. As little as 1

percent of amylose in amylopectin, and possibly as little as 10 percent of amylopectin in amylose, can be detected. This test should prove valuable to chemists for determining the relative amounts of amylose and amylopectin in starch fractions.

Oilseeds

Spectrophotometric data on color provide basis for more efficient grading system for crude green soybean oil

Early frost damages the soybean crop by "fixing" the green pigment of immature beans. As a result, oil obtained from frosted beans is green and, being more difficult to bleach than ordinary yellow oil, sells at a discount. Some years a large acreage of the crop is frosted, resulting in the production of millions of pounds of green oil. This requires changes in processing to remove the color.

Studies are in progress at the Northern Regional Research Laboratory to develop a method for determining the extent of "green damage" in crude soybean oil more accurately than can be done by the method now used in the vegetable-oil trade. The present official grading method, devised in 1942 as an emergency measure, consists in comparing a sample of crude green oil with two standard solutions of nickel sulfate, all viewed through a red filter. By this test the oil falls into one of three grades, depending on whether it is lighter or darker than one or the other standard. A grading system of only three grades for green oil contrasts sharply with the continuous scale for color discount applied to normal oils. A grading system similar to that for normal oils, expressing the extent of green damage on a continuous scale, would indicate the treatment necessary to hold the loss of oil in bleaching to a minimum.

Data on which to base such a grading system were provided by examining oils extracted from several lots of beans collected and frozen at progressive stages of maturity. Color was determined by measurements of the absorption spectra in a photoelectric spectrophotometer, an instrument recently adopted to replace visual methods in the grading of normal oils. There was good correlation between the colors determined by spectral absorption and the grades assigned by visual comparison of the oils with the nickel-sulfate standards obviating any difficulties in establishing new standards. Furthermore, the data showed that it is possible to set up a continuous scale which indicates the amount of pigment that must be removed from a crude oil by refining and bleaching. Further studies will be made to determine the relation of the color of crude green soybean oil to that of the oil after refining and bleaching by methods specified by the trade. Satisfactory methods for evaluating soybean oil and rules based on them are essential for the orderly marketing of the huge soybean crop.

Selection of viable cottonseed for planting speeded by laboratory test

A valuable new test that expedites the selection of viable cottonseed for planting has been provided by the Bureau's Southern Regional Research Laboratory. To obtain this index for a given lot of seed, a representative sample is analyzed for free fatty acid content. The

results serve as a guide to which lots should be reserved for planting. Germination tests are needed on each lot sold as seed stock.

Studies showed that reduction of viability or germination and development of free fatty acid in cottonseed are usually associated with deterioration, whether it occurs in the field or during storage. In general, germination decreases as the free fatty acid in the oil increases. Analysis of individual cottonseed kernels showed that few seeds will germinate if the free fatty acid content of the kernel is above 1 percent, or 3 percent of the oil extracted from the kernel, and that only a relatively small number of kernels have a free fatty acid content between 1 and 5 percent. Evidence of deterioration is not found in every seed of a given lot. The probability that a lot of seed will have a viability above 70 percent lessens rapidly as the free fatty acid content increases. On the basis of these results, it is recommended that cottonseed reserved for planting have less than 0.75 percent free fatty acid in the extracted oil.

This relation between viability and chemical composition was determined by analyzing 254 samples of cottonseed that had been tested for germination by the Seed Laboratory of the Arkansas State Plant Board.

More learned about preparation and reactions of gossypol

New facts concerning the pigments of cottonseed were discovered at the Southern Laboratory. Gossypol, the principal pigment, occurs in the kernel to the extent of about 0.3 to 1.7 percent. It is estimated that as much as 40,000 tons of this pigment could be produced annually in the United States from seed processed for oil and meal. The preparation and properties of gossypol were investigated, with the object of improving the nutritive quality of cottonseed meal and also improving the quality of the oil. An improved method of preparing pure gossypol was developed, and data were obtained on the products formed by the reaction of gossypol with amino acids.

The simple and rapid method devised for recovering gossypol from pigment glands has made possible for the first time the preparation of sufficient quantities of pure gossypol for research on factors affecting the nutritive value of cottonseed meal and on chemical and pharmacological properties of gossypol and related compounds. Gossypol is extracted from fat-free cottonseed flakes with butanone under reduced pressure and isolated as the gossypol-acetic acid complex—a yellow powder. This complex is dissociated with aqueous sodium hydroxide, after which the gossypol is extracted with diethyl ether and recrystallized twice from a mixture of diethyl ether and xylene. Gossypol of a high degree of purity is obtained.

Previous investigators had obtained data indicating that the gossypol molecule has a dinaphthalene nucelus in which all but two of the hydrogen atoms are substituted by aldehyde, methyl, isopropyl, and hydroxy groups. Because of its structure, gossypol is expected to be useful in the production of pharmaceuticals and antiseptics, and possibly as a component of plastics and similar industrial products.

A gossypol-glycine complex was prepared that was nontoxic to small animals, such as mice, rats, and rabbits, even when fed at high levels. The pharmacological and feeding tests were made by the Texas and Georgia Agricultural Experiment Stations, Ralston Purina Co., Swift

& Co., the Southwest Poultry Experiment Station, and the Western Regional Research Laboratory.

Means for estimating fatty acid composition of cotonseed oil aid to industry

Knowledge of the composition of cottonseed oil is important in the selection of oils best suited for making commercial shortenings and salad oils. The Southern Laboratory has contributed to this knowledge by establishing the relation between the fatty acid composition of cottonseed oil and its iodine value, which can be determined easily and quickly by chemical analysis.

Data were obtained on the composition of 48 cottonseed oils of known history. The iodine values of these oils varied over a wider range than any series of oils heretofore available for investigation. The percentages of the fatty acids were correlated with the iodine values. With an increase in the iodine values, the linoleic acids increased, and the oleic and saturated acids decreased. The iodine values of the oils ranged from 88.9 to 117.0. With this variation in iodine value, the linoleic glycerides increased from 34.0 to 56.7 percent, whereas the oleic glycerides decreased from 36.0 to 21.8 percent and the saturated glycerides decreased from 29.7 to 20.7 percent.

The relation between the fatty acid composition and the iodine value affords a ready means for estimating the percentages of linoleic, oleic, and saturated glycerides in cottonseed oils. This information is needed in selecting oils for different uses. For example, oils having the higher percentages of saturated acids are less suitable for winterized salad oils and special shortenings. They give low yields of winterized oils, and shortenings made from them have high melting points.

Poultry, Dairy, and Animal Products

Another crystalline protein isolated from milk

Like other biological tissues, milk contains a complicated mixture of many proteins. Whey, the portion of milk left after the removal of casein, contains 0.6 percent protein, which consists of five main protein components and traces of several proteins with enzymic properties. The purification of three of these components—gamma-globulin, beta-lactoglobulin, and serum albumin—has been adequately described. A reliable method has now been developed at the Eastern Regional Research Laboratory for the preparation of the fourth—alpha-lactalbumin—in the crystalline state in substantial amounts. By sedimentation and also electrophoresis, it was found to be homogeneous. Although this protein was isolated previously, the published method for its isolation has led to failures; consequently its previous isolation has been doubted.

Alpha-lactalbumin, which constitutes about 15 percent of the total protein of whey, is unusual in several respects. It is incompletely coagulated by heat, thus resembling a proteose rather than a typical protein. This finding is of particular interest in connection with the classification of the proteins of milk, since it indicates that the amount of proteose in milk is much smaller than is usually reported. Alpha-lactalbumin has a molecular weight of only 15,000, as determined by sedimentation. It contains 7 percent of the essential amino acid

trytophan and 8 percent of cystine. Its amino acid composition is of particular importance in connection with the nutritional value of milk.

New fatty acid constituents of lard and beef fat discovered

Analyses of lard by ultraviolet spectrophotometric methods developed at the Eastern Regional Research Laboratory produced substantial evidence that lard contains acids of greater unsaturation than arachidonic acid. It is believed that each molecule of these polyunsaturated constituents has five double bonds. No information is yet available concerning the nutritional significance of these compounds, but the proportions found (approximately 0.1 percent) are believed to be too low to affect the physical properties of lard.

Infrared spectrophotometric analysis showed that beef fat contains substantial quantities (5 to 10 percent) of transoctadecenoic acids. These consist mainly of two isomers, elaidic and vaccenic acids. It was concluded that they are not adventitious constituents but important components that may contribute to the unique properties of beef fat.

Research reveals new facts about proteins of egg white

Basic investigations on eggs at the Western Regional Research Laboratory have revealed new facts about proteins of egg white. Because egg white is essentially a solution of these proteins, its biochemical, chemical, physical, and utilitarian properties are the sum of their properties. The uniqueness and the possible significance of these proteins in such diverse fields as embryology, microbiology, enzymology, physiology, and protein chemistry challenge the imagination. There are at least eight known egg-white proteins, and extensive advances have been made in our knowledge of these proteins during the past 10 years. Three of them—ovalbumin, conalbumin, and lysozyme—can be crystallized, and five have unique biochemical properties.

The components of egg white and their important characteristics are—

Ovalbumin, 54 percent, coagulates easily and contains sulfhydryl groups.

Conalbumin, 12 percent, forms complexes with metal ions (iron particularly) and thus retards bacterial growth by iron starvation.

Ovomucoid, 12 percent, combines with the enzyme trypsin and thus inhibits it.

Ovomucin, 1.5 percent, is responsible for the gel properties of thick egg white and has virus antihemagglutinin activity.

Lysozyme, 3.5 percent, attacks cell walls of certain bacteria, causing lysis and death.

Avidin, 0.1 percent, combines with the vitamin biotin and thereby retards growth.

Incompletely characterized proteins, 8 percent, consist apparently of two globulin-type proteins.

Nonprotein components consist of glucose, 4.5 percent, and inorganic salts, 4.5 percent.

Recent studies at the Western Regional Research Laboratory included all the proteins of egg white. In the investigations on the chemical compositions of ovalbumin, conalbumin, lysozyme, and avidin, the chemically reactive groups in the protein molecules responsible for their unique properties were studied. It was shown that ovalbumin contains four sulfhydryl groups (cysteine) per mole rather

than five, as heretofore believed, and that three of the four groups are comparatively reactive in the native protein. It was found that avidin contains nucleotides, but the significance of the association between avidin and the nucleotides was not determined. The integrity of the entire molecule of conalbumin appears to be necessary for its metal-binding properties. As compared with most proteins, conalbumin is unstable to most chemical and physical treatments. In contrast, the enzyme activity of lysozyme is comparatively stable. The ovomucin fraction of egg white was shown to be highly contaminated with other egg white proteins, particularly with lysozyme. Ovomucin of higher purity was prepared, and its gel-forming properties and virus antihemagglutinin activity were determined. Comparative studies showed that thick egg white has four to five times as much virus antihemagglutinin activity as thin white.

Other studies have been concerned with antimicrobial activities of avidin and conalbumin. A large number of organisms are able to grow in egg white in spite of these inhibitory proteins, which bind the iron or biotin essential for the growth of some organisms. In the case of avidin, it was established that the binding of biotin by avidin exists as a measurable equilibrium in which small amounts of biotin are free and thus available to organisms requiring this vitamin. A similar relationship with iron and conalbumin was postulated, and in addition it was found that small amounts of trace elements other than iron (such as copper, zinc, and cobalt) enhance the antimicrobial activity of the protein.

The information obtained in these basic studies is being applied in studies of problems in egg technology. These proteins are used as sensitive indicators of changes that occur in stored shell eggs and in processed eggs, particularly dried eggs.

Foods—General

Toxicity of insecticides present in foods as spray residues investigated

Insecticidal agents occurring in foods as spray residues are part of the larger problem of chemical additives in foods. Chemical additives contribute to better production, better utilization, and greater consumer acceptance of foods. Acute and chronic toxicities and the mechanisms of their physiological effects are the same whether chemicals find their way into foods as insecticides or directly as aids to food processing. During the past year, the Bureau cooperated with the Bureau of Entomology and Plant Quarantine in investigations on the acute and chronic toxicities of dihydrorotenone, allethrin, and the halogenated hydrocarbon, chlordane.

Dihydrorotenone is a derivative prepared from rotenone in an effort to produce an insecticide more stable to light. The acute toxicity of dihydrorotenone was found to be low enough to eliminate the possibility of acute poisoning by foods contaminated with spray residues of this material.

The chronic toxicity of this insecticide was investigated by feeding 9 groups of weanling albino rats a nutritionally adequate basic diet to which dihydrorotenone was added in concentrations ranging

from 10 to 2,560 parts per million parts of the diet. A control group of rats was fed the basic diet only. Rats eating the diets containing 10 and 20 parts per million (p. p. m.) grew normally. Rats that received 40, 80, and 160 p. p. m. grew normally for the first 70 days, but after that they grew at a significantly slower rate. Rats that received 320 p. p. m. were inhibited in growth throughout the experiment, and about half the animals died before completion of the 400-day test period. All rats on diets containing higher levels of dihydrorotenone died in 20 days or less. Although there were no gross anatomical changes in any of the organs and the liver showed no evidence of necrosis, microscopic examination revealed nuclear and cytoplasmic changes in the liver that indicated injury. Periodic examination of the blood revealed no changes in hemoglobin or morphology. Since the amount of spray residue in foods is not likely to exceed a dihydrorotenone concentration of 20 p. p. m., under practical conditions dihydrorotenone appears to be a safe insecticide.

Studies on the toxicity of allethrin are of interest because this insecticide was synthesized by the Bureau of Entomology and Plant Quarantine in an effort to produce an insecticide equal to natural pyrethrins, and make the United States less dependent on imported pyrethrum flowers. The acute toxicities of purified pyrethrins and allethrin were investigated by gastric and subcutaneous administration to rats. No toxic reactions were noted. Cutaneous application to the skin of rats for 30 days produced no local reactions. Impure allethrin caused a temporary skin irritation in albino guinea pigs, but purified allethrin did not cause this irritation. Commercial and purified allethrin were tested for chronic toxicity by long-term feeding experiments with albino rats; the dosage levels ranged from 78 to 5,000 parts of allethrin per million parts of diet. On the highest dosage level, the commercial allethrin caused a slight decrease in rate of growth, as compared with the purified allethrin. Periodic examination of the blood revealed no abnormalities. Histopathological examination of the tissues has not been completed.

The acute toxicity of chlordane was studied by administering the compound to rats orally, cutaneously, and by inhalation. When dissolved in cottonseed oil, the dose of orally administered chlordane required to kill half the animals treated was approximately 590 milligrams per kilogram of body weight. Male rats were slightly more susceptible than females. Daily oral administration of 50 milligrams per kilogram of body weight resulted in toxic symptoms and death. The cutaneous application of about 200 milligrams per kilogram of body weight for 3 or 4 days was fatal to all rats. Rats breathing air passed through chlordane showed no signs of toxicity after exposure for 120 hours.

The chronic toxicity of chlordane was investigated by feeding groups of rats diets that contained 10 to 640 p. p. m. of the insecticide. Rats that received 320 p. p. m. or more of chlordane showed definite retardation of growth, although the effect was less marked with female rats than with males. No abnormalities of the blood were found. At time of autopsy, all rats that received 80 or more p. p. m. of chlordane had enlarged livers, and microscopic examination showed enlargement of the liver cells and their nuclei and nucleoli.

FUNCTIONAL ORGANIZATION OF THE BUREAU

The Bureau of Agricultural and Industrial Chemistry is organized on a decentralized basis. Its research is conducted at four large regional research laboratories—located at Wyndmoor, Pa., Peoria, Ill., New Orleans, La., and Albany, Calif.—and at several smaller specialized branch stations. The Chief of the Bureau and his immediate staff, including five Assistant Chiefs, are located in Washington, D. C. The Assistant Chiefs are assigned responsibility for particular phases of the Bureau's activities. There are four Regional Directors, each of whom is responsible to the Chief of the Bureau for all research in his region. The organization of the Bureau is as follows:

Chief of Bureau	G. E. Hilbert.
Assistant Chief, utilization of cereal and forage crops, sugar, and special plants.	C. F. Speh.
Assistant Chief, utilization of oilseeds, and poultry, dairy, and animal products.	G. W. Irving, Jr.
Assistant Chief, utilization of fruits and vegetables	J. R. Matchett.
Assistant Chief, utilization of cotton and other fiber crops, and agricultural residues.	W. M. Scott.
Assistant Chief, administration	H. A. Donovan.
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