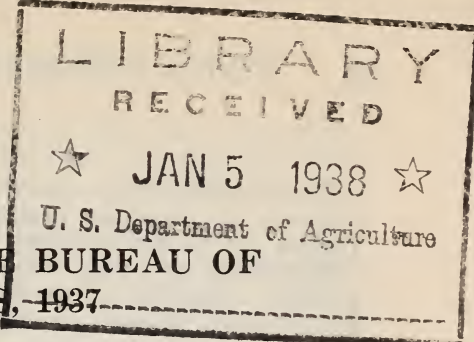


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**REPORT OF THE CHIEF OF THE BUREAU OF  
CHEMISTRY AND SOILS, 1937**

UNITED STATES DEPARTMENT OF AGRICULTURE,  
BUREAU OF CHEMISTRY AND SOILS,  
Washington, D. C., August 31, 1937.

HON. HENRY A. WALLACE,  
*Secretary of Agriculture.*

DEAR MR. SECRETARY: I present herewith the report of the Bureau  
of Chemistry and Soils for the fiscal year ended June 30, 1937.

Sincerely yours,

HENRY G. KNIGHT, *Chief.*

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## INTRODUCTION

The production of agricultural materials through the medium of plant growth depends on chemical processes, energized by sunlight, in which the raw materials are carbon dioxide, oxygen, and nitrogen of the air and numerous soil constituents including water, inorganic compounds derived from minerals, and organic compounds formed through the action of micro-organisms on the products of former plant and animal life. Whether or not these chemical processes go on depends not only on the availability of energy, but also on the availability of raw materials. Since the gases of the atmosphere and sunlight, in most places and at certain seasons, are always sufficiently abundant, it is the availability of raw materials from the soil which determines the course and efficiency of nature's chemical processes that result in agricultural plant and animal products. One of the most important of these raw materials is water, the availability of which in suitable quantity depends primarily on the physical and chemical properties of the soil colloids which make it possible for soils, in varying degree, to retain water for fairly long periods after wetting. The properties of their colloids also make it possible for some soils to retain, in spite of frequent wetting with excessive quantities of water, inorganic and organic plant foods, naturally present or added as fertilizers, and to supply them, as needed, to growing plants.

The kinds and quantities of raw materials suitable for the production of plants vary in different soils and, consequently, the kinds and quantities of plants that different soils can produce vary. Some soils contain substances which interfere with the natural chemical processes involved in plant growth and thus produce abnormal or unhealthy plants or alter the resulting products so that they are not suitable raw materials for the chemical processes involved in animal growth or sustenance. Moreover, some soils are deficient in one or more of the raw materials required for the normal course of the chemical processes involved in the production of certain plants. Such deficiencies may prevent the production of particular plants entirely, they may result in abnormal or unhealthy plants, or they may so alter the composition of the resulting products that they cause unhealthy conditions in animals consuming them as food.

Food is one of the prime necessities of human and animal life, and the soil is the principal source of food. The agricultural utilization of soils results in many products that contribute to the needs and desires of human beings, but its primary purpose is, and probably always will be, the production of food. The composition of foods and their utility for maintaining human and other animal life in a state of health depends on the composition of the soil. The abundance of these food products depends largely on the abundance of water and plant foods in the soil, which, in turn, depends in large measure on the ability of the soil colloids to retain them.

The Bureau of Chemistry and Soils is a research organization engaged in the application of chemistry and related sciences to the study of soils, soil-amendment materials, and soil products; and it is, therefore, concerned with the fundamental things in agriculture. On the basis of the scientific knowledge gained in these studies, soils are classified according to origin, characteristics, and usefulness for various agricultural purposes, and the distribution of the various classes and types of soils is shown on maps of counties or other comparatively small areas. The existence of deleterious substances in certain soils and their effects on the composition and utility of plants is noted, as well as deficiencies of certain substances necessary for vigorous and healthy plant growth and the need for special fertilizer or other soil-amendment materials.

Conditions contributing to the maintenance or increase of soil fertility through the nitrogen-fixing and other chemical activity of micro-organisms and through the addition of inorganic and organic plant foods are studied; also the suitability of various substances and mixtures of substances for correcting soil deficiencies, and economical methods for producing efficient fertilizers from available raw materials.

Plants and plant products are studied both with regard to composition and as to how their composition and utility for food and other purposes are affected by the composition of the soil. The influence of feeds on the composition and properties of certain animal products is also studied.

Means for preventing deteriorative chemical changes in agricultural products, due to enzyme action, micro-organisms, and other agencies, are investigated. Much attention is given to the best utilization of agricultural products through

chemical processing. Studies are made on their use for new and better food products and on the use of inedible products and byproducts in chemical industries. Nonfood uses through chemical processing are being sought for food crops produced in excess of the needs for food purposes.

The results of the work of this Bureau during the year, along these various lines, are reported briefly in the following pages. Further information concerning many of the subjects discussed may be obtained from the 274 publications emanating from the Bureau during the year and listed at the end of this report.

## CARBOHYDRATE INVESTIGATIONS

### SUGARS, SUGARCANE, AND SUGAR BEETS

Investigations on the chemistry and technology of the production of cane sugar were carried on at the Houma, La., field station, in cooperation with the Bureau of Plant Industry, and also at the Audubon Sugar School in Baton Rouge, La., where improved working facilities became available during the year through cooperative arrangements with the College of Pure and Applied Science of Louisiana State University.

Studies pertaining to the production of direct-consumption sugar of improved quality and byproduct molasses were undertaken. A result from this work was the development of a process of reducing the sulphite content of edible molasses by the use of chlorine, thereby improving its quality and marketability. A public-service patent (No. 2,043,911) covering this process has been granted.

In the production of raw sugar in Louisiana, it is customary, when crystallization facilities are lacking, to allow the second molasses, after it is boiled to a heavy density, to crystallize for several months before separating the final molasses. Frequently this separation is extremely difficult to accomplish, and the reasons for this are quite obscure. A fundamental study of the manufacture of this magma sugar was, therefore, initiated. Clarification of the juices prior to crystallization doubtless has a very important influence on the workability of the massecuites in the vacuum pan and centrifugals, and considerable attention was given to a study of the clarification characteristics of the juices from different varieties of cane and to improving the clarification of juice by the development of mud-separating devices, as well as to efficient processes of sugar crystallization.

Studies in past years have shown that the juice of each variety of sugarcane tends to have definite characteristics with respect to behavior during clarification. From analytical and clarification studies on the same juices it appeared that these characteristics of individual varieties depend on the kinds and quantitative relationships of nonsugar substances present, and that the natural tendency of a variety may be intensified or minimized by conditions of growth. Recent work has shown that different fields of the same plantation, when planted with the same variety, produce cane of decidedly variable composition with juices of very different clarification qualities. During the last season a special study was made of the variety Co. 290 which formed the major part of the crop and was grown almost exclusively in certain localities. A clarification procedure yielding juice of pH 7.0 or above was found necessary for good clarity. However, since the mud volumes are large and the high content of salts characteristic of this variety adversely affects sugar boiling under neutral or alkaline conditions, it was concluded that a procedure yielding clarified juice of pH ranging from 6.5 to 6.7 results in better average operating condition.

Comparable samples of five of the newer commercial varieties of sugarcane from six different test fields were studied with regard to their nonsugar content and the composition of their juices, in order to determine their relative sugar-making values. These included CP 28-11, CP 28-19, Co. 281, CP 29-320, and CP 29-116, the last having been released for cultivation in 1936. The crusher juice from each sample and also the whole mixed juice from each variety was analyzed for total ash, phosphates, sulphates, chlorides, potash, lime, magnesia, and silica. CP 28-11 was characterized by high ash and high sulphate content, CP 28-19 by low ash content, CP 29-116 by low ash and low lime content, and Co. 281 by high lime and high sulphate content. A number of new cane seedlings also were studied in a preliminary way. Incidental to this work, a new method was devised and used for estimating the approximate total salt content of cane juices.

Data were obtained showing that there are striking differences in the distribution of nonsugars between the nodal and internodal portions of different varieties of cane. In Co. 290 the salts are mostly soluble and concentrated in the internodes, whereas in Co. 281 the concentration is greatest in the nodes. This difference in distribution affects the extraction of salts at different stages of milling and thereby the reduction factors.

The cane varieties Co. 290, Co. 281, CP 807, and CP 28-19, as well as certain seedlings, were compared on the basis of whole cane analysis, as contrasted with the substances extracted by milling and those extracted by diffusion in water. The low nitrogen content previously found to be characteristic of the juice of variety Co. 290 was found to hold true for the whole millable cane. Analysis of the whole mill juice gives information on the quantities of phosphate and potash removed from the soil.

Studies were continued, in cooperation with the Division of Sugar Plant Investigations, Bureau of Plant Industry, on the causes and prevention of deterioration in harvested sugarcane during short-time storage. This is an important problem because, if it were possible to store sugarcane without appreciable deterioration, it would allow the operation of the sugar factory during the normal rainy periods of the harvesting season and prevent a large loss from freezing, provided the cane were stored prior to the freeze. In previous studies it had been noted that, under high humidity, a temperature between 55° and 65° F. was best for preventing deterioration, and that the rate of deterioration increased below and above these limits of temperature. In these studies 45° F. was the lowest temperature employed. In this year's studies, however, cane was stored at a minimum temperature of 36° F., and it was found that the rate of deterioration was less than at 45°, probably because of reduced physiological activity. Previous findings that storage under low humidity, which allowed loss of moisture from the cane, was conducive to loss of sucrose by inversion were substantiated.

Tests on a large number of CP seedling varieties of cane available at the Houma station, to determine their suitability for windrowing, which must frequently be resorted to in order to prevent heavy losses of the crop by freezing, revealed that several were equal or superior to Co. 281 in this respect. This knowledge is useful in the final evaluation of canes that otherwise might be suitable for commercial planting, and it indicates crosses which might be made in breeding experiments to bring out desirable characteristics in cane.

Further evidence was obtained that the inversion of sucrose in harvested sugarcane under certain conditions of storage is not proportionate to the quantity of invertase present, and that inversion is controlled by some unknown factors, other than the mere presence of invertase.

As a result of the Bureau's work on beet sugar, in cooperation with the beet-sugar industry, there has been continued progress in the quality and uniformity of this important domestic product. Objections formerly raised by consumers to the use of beet sugar for certain purposes have been largely eliminated, and this commodity should enjoy its rightful parity with cane sugar and should find greater consumption in its own territory with substantial savings in freight charges. Composite sugar samples, representative of all the beet-sugar-producing areas of the United States, were subjected to detailed analysis. There was a marked degree of uniformity in this year's production. The results are fully discussed in the annual beet-sugar report issued by the Carbohydrate Research Division.

During the year further study was made of methods for the determination of the color and turbidity of sugar solutions and of the appearance of granulated sugar by means of the Brice-Keane photoelectric reflectometer which was developed in the Department. A simplified procedure for these determinations will be described in a forthcoming publication. The Brice-Keane reflectometer is now a commercial instrument and is in use in the beet-sugar industry for the purpose of obtaining an index of the appearance and quality of the sugar produced.

The sugar arabinose was prepared from sugar-beet pulp. This sugar and xylose were used for the preparation of fondant. The results show that both arabinose and xylose can be put in the solid phase of the fondant. Arabinose has the advantage over xylose in being less soluble. A study was made of the separation of dextrose and levulose in invert-sugar preparations, on the basis of the difference in their solubility in various solvents, but an entirely suitable process has not yet been developed.

## FARM-MADE SIRUPS

On many thousands of farms in the South, particularly the smaller ones, sugarcane is grown for the production of sirup instead of sugar. The crop is harvested and made into sirup late in the year when other crops have been harvested and farm labor otherwise would be idle. The quantity of sirup made on individual small farms ranges from 50 to 500 gallons a year, and the total annual production amounts to between 15 and 20 million gallons. Sugarcane for sirup is a diversification, subsistence, and small cash crop.

The Bureau's work on sugarcane sirup during the last year, designed to improve efficiency of production and quality and uniformity of product for both farm consumption and profitable sale, was in cooperation with the Department's Extension Service, the Works Progress Administration, and groups interested in community plants. These organizations and groups were given advisory assistance in the design and operation of plants and in the use of recently improved methods.

Nearly all of the sorgo sirup, about 17 million gallons of which are produced annually in the United States, is made by farmers with extremely limited facilities. Much of the sirup is of inferior quality and is sold at prices which yield little profit to the farmer. To aid in the correction of this situation, the Bureau is studying means for improving the quality and uniformity of farm-made sorgo sirup. Processes for improving the quality of farm-made sirups must be practicable and embody such features as low cost of equipment and simplicity of operation, if they are to find ready application. The malt diastase process for the prevention of jellying and consequent slow boiling or scorching and the practice of topping the stalks to the fifth internode to reduce the mineral, acid, and starch content of the juice fully meet the limitations of farm-scale sirup production. Studies conducted in cooperation with the Mississippi Agricultural Experiment Station during the year showed that further improvement in quality of sirup could be accomplished by combining these two methods. The details of these methods and descriptions of other improved practices and equipment were incorporated in Farmers' Bulletin 1791, entitled "Farm Production of Sorgo Sirup", which is in press.

Cooperative work in Mississippi on the composition of juices and quality of sirup from different sections of the stalks, in which 8 additional varieties of sorgo cane were used and about 100 samples of sirup were made, confirmed the conclusions from last year's work that the farm value and marketability of sorgo sirup can be materially improved by cutting off and discarding several joints of the stalk from the top. The discarded top joints can be well utilized as feed for livestock, and the comparatively small reduction in yield of sirup is more than compensated by the improvement in quality of the sirup and the feeding value of the discarded top sections of the stalks.

The Bureau continued to cooperate with the State extension services of Mississippi and Alabama, which demonstrated to farmers the improved methods of production of sorgo sirup developed by the Carbohydrate Research Division. In Alabama, these improvements have been incorporated in model community sirup plants where successful demonstrations were given throughout the season.

Additional master color standards for grading maple sirup were prepared for State agencies in producing areas, and these agencies in turn prepared color standards for distribution to farm producers. The object of this service is to correct lack of uniformity in quality grading and to enable farm producers to determine independently the quality of the sirup they offer for sale.

## HONEY

The decline in the consumption of honey during recent years has reduced the income of beekeepers, of whom there are about 800,000 in the United States with about 5,000,000 colonies of bees. The value of the beekeeping industry resulting from cross-pollination of the blossoms of orchard, garden, and field crops is probably 10 times the value of the honey and beeswax produced. Marked curtailment in beekeeping, which might result from a continued decline in the consumption of honey, would be a serious threat to the future adequacy of insect pollination of agricultural crops. Even when bees are kept specifically for pollination in fruit orchards, profitable disposal of the honey is a vital consideration.

The development of potential industrial outlets for honey would stabilize the beekeeping industry. There is evidence that a number of industries would

like to use honey, and that the failure of some to use this product satisfactorily has resulted from a lack of knowledge concerning the composition and properties of honeys of different floral types and their relation to the requirements for particular uses. The Bureau, at the urgent request of national organizations interested in the production of honey, is working to acquire and disseminate this knowledge.

One of the most valuable properties of honey is its ability to absorb and retain moisture. This is particularly characteristic of honeys containing high proportions of levulose and colloidal organic substances. A study of the ability of honey to absorb and retain moisture in comparison with that of other saccharine liquids has led to the following conclusions: (1) Honeys of high colloid and nonsugar contents exhibit higher hygroscopicity than honeys of low colloid and nonsugar contents, (2) when crystallization occurs in honey its hygroscopicity is materially altered, (3) honeys in general are more hygroscopic than other saccharine liquids having the same moisture content, and (4) honeys of certain types used in baked goods (particularly cakes) increase the retention of moisture of these products, improve their quality, and retard staling. These observations indicate that if honeys of suitable characteristics are selected their use in baked products has definite advantages. The property of absorbing and retaining moisture, which honeys of certain types possess in such pronounced degree, may also give honeys of these types special advantages for use in industries other than baking.

The market quality of extracted honey has been greatly improved during the year through the widespread adoption of the Bureau's new method of processing, which depends on rapid filtration under conditions that insure practically perfect retention of flavor and yield a product of brilliant clarity, having very little tendency to granulate. A public-service patent (No. 2,070,171) covering this process for purifying extracted honey was issued on February 9, 1937.

#### STARCHES

In the United States there is an annual demand for about 300 million pounds of root starches for industrial uses. This demand is now largely supplied by importations of cassava and potato starches. By utilizing the surplus and culls from a number of starch-bearing crops grown in this country, a substantial part of this special starch market could be supplied with domestic products, with material benefit to our own agriculture. The Bureau, therefore, is giving attention to the chemistry and technology of producing and utilizing root starches.

Primary consideration has been given to the sweetpotato as a source of commercial starch and derived products, because of its suitability for cultivation over extensive agricultural areas and because there is always a large supply of culls and frequently a surplus of the crop. Continued work on sweetpotato starch during the year resulted in the discovery of a simple, cheap, and efficient method for obtaining high-grade commercial starch from sweetpotatoes. This method, which is a marked improvement over the one in previous use, was used exclusively during the 1936 operating season (October and November) at the Laurel, Miss., sweetpotato-starch plant, where 420,000 pounds of starch and 178,850 pounds of dry pulp feed were produced successfully on an industrial scale. Further study was made of the suitability of sweetpotato starch for utilization in industries, such as paper, textile, laundry, and dry cleaning, and it was concluded that sweetpotato starch is superior in certain respects to other starches used in these industries.

During the year The Chemical Foundation, Inc., maintained three fellowships, under the supervision of the Bureau, for studying the utilization of sweetpotato starch as a raw material for the production of more valuable compounds or derivatives and the sweetpotato-storage problem. The methods employed in storing sweetpotatoes for the food market are not satisfactory from the point of view of starch making because of the rapid loss of starch in addition to the high cost of handling and curing the crop. The study of storage problems resulted in the discovery of a method whereby it is possible to dehydrate sweetpotatoes at low cost and to obtain a stable product that can be converted into starch or into a flour suitable for certain industrial uses. The novel feature of this method is a chemical treatment of the sliced or ground sweetpotatoes, which causes the tissues to plasmolyze so that the juice can be pressed out much more effectively and the residue dried much more quickly and efficiently. By using the dried product, which is stable and can be stored indefinitely, sweetpotato-starch factories could operate during the entire year instead of



only 2 or 3 months in the fall. In large-scale operations, the soluble sugars in the expressed juice possibly could be converted into alcohol or feed at sufficient profit to pay the cost of dehydration.

### CELLULOSE

Basic research on the chemistry of cellulose and its compounds was conducted by the Carbohydrate Research Division during the year in an investigation of the hydrolytic products of cellulose. The degradation of cellulose by methyl alcohol-hydrochloric acid was studied in a preliminary way. Identification of the soluble products has not yet been completed. They are non-reducing, optically active substances very susceptible to caramelization. Probably those obtained thus far are mixtures of the glycosides of dextrose and cellodextrines. To verify this supposition glycosides were prepared, starting with commercial dextrose which required preliminary purification. A study of the reactions of the glycosides of dextrose in comparison with those of the degradation products of cellulose under investigation is under way. Octaacetyl collobiose was prepared by acetolysis, and its hydrolysis by alcoholic potassium hydroxide and by alcoholic ammonia was studied.

### MISCELLANEOUS CARBOHYDRATES

The carbohydrates found in vegetation range widely from the simplest sugars to the most highly complex polysaccharides. Because of the abundance of some carbohydrates in certain plants, these plants have become of greatest economic importance in agriculture as sources of food or industrial products. Thus, sugarcane and sugar beets are grown for their sucrose; corn and potatoes for food and as raw materials for industrial products, chiefly because of their starch content; and cotton primarily for its seed hairs which are almost pure cellulose in a form eminently suited for use in textiles. There are other plants which store inulin, instead of starch, as their reserve carbohydrate. This compound, on hydrolysis, yields levulose (fructose) in contrast to dextrose (glucose) from starch or cellulose. This and similar compounds have been studied little with regard to their suitability for use in the diet or for industrial products, and increased knowledge of the chemical identity and properties of these carbohydrates should lead to a more profitable utilization of some of the minor crops as well as of plants which could be grown as crops in agricultural diversification programs. The Carbohydrate Research Division has given attention to some of these plants and has published information concerning them in Miscellaneous Publication 237, entitled "Food Plants of the North American Indians."

Progress was made in perfecting a process for the production of levulose sirup from the inulin in chicory roots, but primary consideration was given, in the investigation of miscellaneous carbohydrates of domestic plants, to a basic study of plant hemicelluloses. Data were obtained on the effects of concentration, time, and temperature on the extraction of hemicelluloses from sugar-beet pulp with acid and alkaline solutions.

## FOOD RESEARCH

### CITRUS FRUIT PRODUCTS

In the Los Angeles laboratory, sections of citrus fruits, including navel oranges, tangerines, grapefruit, blood oranges, and various citrus hybrids, were packed in sirup to determine their suitability for use in salads. The filled cans were sterilized for 6 or 7 minutes in water at 195° F., while in motion. Some of the citrus hybrids had a better flavor than the Japanese canned mandarin oranges being sold in this country. It was found that canned blood oranges hold their color, and it is believed they will make a good salad material. Storage tests are still in progress.

Tangerine juice was prepared for canning by cutting the fruit in half across the sections and then pressing the halves between stainless steel rolls so adjusted as to cause minimum crushing of the peel. The juice was then screened, deaerated, and flash-pasteurized. The product contained 0.02 percent of volatile oil which gave it the characteristic tangerine flavor lacking in reamed juice. This quantity of oil may cause subsequent off-flavor, but the juice has not been stored long enough to determine whether or not this will occur.

In experiments on the cold storage of Florida orange juice in bulk, pasteurized and raw juices were stored in covered, but not airtight, 5-gallon con-

tainers at temperatures of 32° to 34° F. At intervals, plate counts of bacteria were made on the juice and tests were made of the potency of vitamin C. All samples showed the beginning of off-flavor after 6 weeks of storage, and all had fermented within 8 weeks. The pasteurized samples fermented as quickly as the raw samples. The vitamin C content showed a decrease, ranging from 19 to nearly 32 percent, in 4 weeks under the conditions of storage.

Studies on the vitamin C content of commercially deaerated and flash-pasteurized orange juice packed in glass bottles showed that there was a loss of vitamin C during storage, which amounted to about 19 percent at the end of 11 months.

A public service patent (U. S. No. 2,060,242) was obtained for a new type of deaerator which is more efficient than that used heretofore. One of these new deaerators of commercial size is under construction by a manufacturer of food machinery for use in a new cannery being built in Florida for the purpose of packing citrus juices by deaeration and flash pasteurization.

Tangerine concentrates were prepared which contained, in some instances, as high as 68 or 72 percent of total solids. When diluted with water they lacked the tangerine flavor, but the addition of an emulsion of tangerine oil improved the product to some extent.

Unsuccessful attempts were made to hydrolyze waste from the grapefruit canneries and to ferment it with selected organisms, in order to yield butyl alcohol, acetone, butyric acid, and lactic acid. Attempts to preserve grapefruit peel by brining have been unsuccessful thus far. The disposition of semi-solid wastes from grapefruit canneries has become an important problem. These wastes are crudely screened and poured into nearby lakes. Preliminary experiments have been made on the treatment and filtration of cannery wastes to render them innocuous.

With 29 canneries in Texas packing canned grapefruit juice, the chemists at the Weslaco laboratory spent a great deal of time giving advice as to the design and installation of equipment and assisting in the solution of problems arising during the operation of new plants by inexperienced canners.

Dextrose was found to be better than sucrose for use in canning tender segments of pink Marsh and Duncan grapefruit. For comparison, 1½ ounces of either dextrose or sucrose or 2½ fluid ounces of either dextrose sirup or sucrose sirup, prepared with water or juice, were added to each No. 2 can of segments. The packs prepared with 2½ fluid ounces of 50-percent dextrose sirup a can were among the best as regards appearance and flavor. Each pack showed some variation in individual cans, but all were satisfactory from the commercial point of view.

The results of storage tests on grapefruit beverage bases showed that vacuum-concentrated bases and bottlers' sirups can be stored satisfactorily for more than 2 years at temperatures below 45° F. Finished carbonated grapefruit beverage has been stored for more than 2 years at ordinary temperatures without spoilage or sufficient deterioration to render it unmerchable.

It is interesting to note the statement in the July 1937 issue of *The Canning Age* that the entire citrus juice output of Texas is handled through a flash-pasteurization method. The details of this method were worked out largely by the Weslaco, Tex., and Winter Haven, Fla., laboratories of the Food Research Division of this Bureau.

#### DECIDUOUS FRUIT PRODUCTS

In canning investigations at the Los Angeles laboratory, Boysen dewberries were packed in 40-percent sirup in No. 2 cans of three types, namely, ordinary lacquer on coke tin plate, citrus enamel on coke tin plate, and citrus enamel on charcoal tin plate. A comparison was made of storage at room temperature and at 38° F. At the end of 11 months the pack stored at 38° had slightly better color and flavor than those stored at room temperature. The pack giving best results was one in charcoal tin-plate cans coated with citrus enamel. The fruit had been covered with 40-percent sirup, closed cold under 25 inches of vacuum, and cooked for 10 minutes at 212° F.

Examination of the 1935 pack of Young dewberries after 14 months of storage showed that no springers or swells had developed in any of the coke tin-plate cans coated with ordinary lacquer or in the charcoal tin-plate cans coated with enamel.

In the laboratory at Winter Haven, Fla., a strawberry sirup was prepared by crushing fresh cull berries, adding corn sugar and cane sugar to 64° Brix, and then filtering clear. Sodium benzoate was added as a preservative because the

sirup was not stored in sealed containers. After storage at room temperature for 1 year, the product still retained its strawberry aroma and taste. One part of this sirup diluted with 4 parts of water and carbonated at 2.5 to 3.0 volumes made a very satisfactory beverage, but during storage at 40° F. for 4 months this beverage acquired a musty flavor. Assuming a cost of \$40 a ton for cull strawberries, the sirup would cost about 39 cents a gallon.

Papaya slices and pulp were canned experimentally. When processed longer than 40 minutes at 240° F. the material had a caramellike taste. Since spores of *Clostridium botulinum*, if present, probably would not be destroyed by heating at 240° for 40 minutes, it was considered advisable to give the product a decidedly acid reaction. This was accomplished by adding citric acid (0.8 g per 100 g of pulp), which gave the product a pH of 4.0. Sugar, at the rate of 15 g for every 100 g of pulp, was added to counteract the sour taste. No. 1 cans filled with the pulp were exhausted for 20 minutes at 212° F., sealed, and then processed for 45 minutes at the same temperature; others were vacuum sealed at room temperature and processed for 65 minutes at 212°. In the case of slices, a 6° Brix sugar solution having an acidity of 0.4 percent was added. After processing, the product had a pH of 4.3. Some No. 1 cans were vacuum sealed at room temperature and processed for 30 minutes at 212°; others were exhausted for 5 minutes at 212°, sealed, and processed at 212° for 30 minutes. Further work is planned in this field. One citrus cannery planned to make an experimental pack of papaya slices during the 1937 season. Analyses have been made of papaya juice and papaya seeds.

Under the supervision of the Seattle, Wash., laboratory, frozen packs were made of 147 field selections of promising seedlings and hybrids and of named varieties of strawberries, about 35 selections of blackberries, and about 50 selections of raspberries, in cooperation with representatives of the Bureau of Plant Industry and the fruit products industries department of Oregon State College at Corvallis, Oreg. In cooperation with the Western Washington Experiment Station, 12 named and 2 seedling selections of blueberries and 58 selections of raspberries, grown at the station, were packed by freezing.

At the Pullman, Wash., laboratory very satisfactory table sirups were prepared from Delicious apples of low acidity by concentration, both in the rapid evaporator and in the vacuum pan. Recovered esters were combined with the concentrated juice to give a desirable apple flavor.

In a study of the suitability of western-grown apples for the commercial production of canned apple sauce, it was found that sauce made from western varieties of apples is more yellow than that made from eastern apples. In general, western varieties are less tart than eastern varieties. By selecting the most tart varieties, adding less sugar than is customary in the East, and guarding against lowering of acidity by dilution, satisfactory sauces were made. Yellow Newtown, Grimes Golden, Esopus Spitzenberg, White Pearmain, and Northwestern Greening proved suitable for this purpose.

Six varieties of northwestern-grown peaches were studied in a preliminary way with regard to their suitability for commercial canning. It is planned to extend this work both as to varieties and methods adaptable to commercial use. The canning of freestone peaches has become of commercial importance in the Pacific Northwest, where the fruit is not picked until soft ripe when full flavor has been attained. The customary commercial method of peeling by treatment with lye cannot be used, because the texture of the soft fruit is broken down, giving a ragged appearance to the product.

A fruit-juice concentrator of radical design has been constructed and used for preparing concentrated juices from apples, pears, and cherries. This equipment consists of a steam-jacketed stainless steel tube in which the juice is heated while flowing. The tube is connected to a flash pot maintained under vacuum. The juice emerges into this pot in the form of a mixture of vapor and slightly concentrated juice. Since the sensible heat of the juice at the time of emergence is not enough to bring about sufficient concentration in one passage, the juice is recirculated until the desired concentration is reached. The vapor from the evaporation of the juice passes first to a warm condenser, where water is separated from the more volatile flavoring constituents which pass over to a second condenser maintained in an ice bath. The volatile flavoring constituents thus collected are combined with the concentrated juice.

Two methods of pasteurization have been found satisfactory for apple juice. The juice may be flash pasteurized in steam-jacketed, ribbon coils of the type developed for citrus juices or it may be pasteurized in No. 2 tins after vacuum sealing by immersing in boiling water for 6 minutes and then cooling. No cooked taste is developed by either of these methods.

Apple juice was canned in 10 different types of tinned containers of various weights of plate coated with different protective enamels. Containers coated with enamels developed for beer and for wine have proved the most satisfactory. In unlacquered cans the juice bleached and formed a cloudy precipitate.

#### VEGETABLES AND VEGETABLE PRODUCTS

Experimental packs of frozen lima beans, spinach, kale, peas, green beans, and sweet corn prepared by the Bureau of Plant Industry, in 1934 and 1935, were examined to provide a standard for any future critical judging of such frozen products. The numbers and types of micro-organisms surviving the freezing and storage period at freezing temperatures were determined.

At the Los Angeles laboratory, several varieties of commercially grown cauliflower and broccoli were used in a study of the technology of preserving vegetables by freezing. Preliminary observations were made which may be of use to the California Agricultural Experiment Station in making selections for 1938 plantings on experimental plots. Experiments were also made on the freezing preservation of asparagus. An exhibition of material frozen at the Seattle laboratory was made at the Los Angeles laboratory in April. Peas, lima beans, snap beans, and sweet corn were shown, together with some cauliflower frozen at Los Angeles. About 30 persons were present when the cans were opened, including local ice and cold-storage men, county and State extension service representatives, women from the nutrition department of the University of California at Los Angeles, agricultural writers, cauliflower and asparagus growers, packers of vegetable juices, makers of bakers' supplies, seed growers, and local and State representatives of the Agricultural Adjustment Administration.

A mimeographed preliminary report of results obtained by the Seattle laboratory in a study of the quality of frozen vegetables as influenced by variety was prepared jointly with the representative of the Western Washington Experiment Station and with the collaboration of the Bureau of Plant Industry and the Washington Agricultural Experiment Station. This report, entitled "Vegetable Variety Trials in Relation to Freezing Preservation," gives the results of the cooperative work on the suitability of vegetable varieties grown during 1936 for freezing preservation. About 500 copies have been sent out in response to requests from canners and packers, seedsmen, distributors, and others interested in the subject.

At the Pullman, Wash., laboratory, tomato juice was prepared by deaeration and flash pasteurization with the expectation that this process would yield a product of better flavor and color and also of higher vitamin C content than that obtained by the usual commercial processes. In two instances out of three, samples of deaerated tomato juice were found to have a higher ascorbic acid (vitamin C) content than controls not deaerated. Further work is necessary to obtain consistent results and to determine conditions under which deaeration can be used to obtain a product of higher vitamin C content.

At the Weslaco, Tex., laboratory, canning experiments were made with several varieties of English peas and with string beans, whole tomatoes, and tomato juice. This work was largely of a preliminary nature but was expected to provide a basis for plans for extensive studies in 1938.

Studies on the commercial fermentation of cucumber pickles under southern climatic conditions have been continued at State College Station, Raleigh, N. C., in cooperation with the North Carolina Agricultural Experiment Station. Results obtained in experiments on the salting of vats of cucumbers indicate that low brine concentrations can be used effectively under southern climatic conditions, and that it is not necessary to use very high concentration to prevent spoilage. Extensive chemical analyses were made during the year on fresh material and salt stock. The reduction in sugar content of the brines roughly corresponded to the increase in lactic acid content during fermentation. It was found that the maximum production of acid was attained in 5 or 6 days, and that a yeast fermentation often followed after a few days. This yeast fermentation gave rise to so-called boiling of the vats. The gas given off was about 99 percent carbon dioxide.

A pack of fresh cucumber pickles was put up and pasteurized at 160° F. for 15 minutes. The pickles retained their crispness, color, and flavor. After 8 months of storage at room temperature they were still in good condition.

A number of barrels of cucumbers with added dill were salted, using various salt concentrations. Organic acids were added in different amounts to some of the barrels. The best dill pickles produced were those in which acetic acid had

been used. These experiments are being repeated, in order to determine the correct salt concentrations and acidities necessary to produce high-quality dill pickles under southern conditions.

#### STALING OF BAKERY PRODUCTS

It was found that staling of bread is not retarded simply by preventing the loss of moisture. Bread made with 85 percent of wheat flour and 15 percent of rye flour had better keeping quality than ordinary wheat bread, and the addition to wheat flour of soybean flour low in fat appeared to slightly retard staling of the bread. Bread and pound cake were stored at subfreezing temperatures for considerable periods with satisfactory results. Bread stored at 0° F. retained its freshness better than similar breads stored at 20° or 30° for the same length of time. Bread taken directly from the oven and placed in a metal container with a tight-fitting top was stored at 0° for 49 days. After being thawed, its qualities were similar to those of bread 1 day old. Pound cake, after having been stored at 0° for 6 days and then thawed out, seemed like fresh cake. Even after such cakes had been frozen for 30 and 69 days, they were judged equal in freshness to an unfrozen cake 1 day old.

#### GLUTEN BREAD

In experiments on the use of wet gluten in breadmaking, it was found that remixing of the fermented dough, just before panning, improved, to a marked degree, the grain and texture of the loaf. It was also found that soybean flour, rye flour, other flours, or combinations of other flours, could be added to the gluten to make a specialty or distinctive bread of high protein content of excellent quality. The use of wet gluten, prepared by washing most of the starch from wheat-flour dough, should make it possible to bake a very pleasing combination of high-protein, low-starch bread at only slightly higher cost than that of ordinary bread.

#### MINERAL CONTENT OF FOODS AND FEEDS

A study of the acid-base balance of cereals and some related food materials showed that practically all common cereals yield a slightly alkaline ash, but that all have an acid balance. Buckwheat, popularly but erroneously classed as a cereal, has a slight base balance. Jack beans, soybeans, and potatoes have a distinctly alkaline ash and a distinct base balance. The acid-base balance of foods may not only influence the acid-base equilibrium of the animal and human systems, but may also have an effect on the end products of mineral metabolism.

#### RANCIDITY

Further attention was given to the relation of peroxide content of vegetable oils and foods containing fats or oils to the development of rancidity. Both rancidity and the formation of peroxides were previously found to result from exposure of vegetable oils to ordinary sunlight. Irradiation of oils with light of constant intensity (from 500-watt CX Mazda tungsten filament lamps) in glass transmitting the ultraviolet rays led to the following conclusions: (1) Peroxides increase at a more uniform rate when oils are irradiated with light of constant intensity than when irradiated by sunlight; (2) when an oil which has been protected by a sextant green filter and already has developed a high peroxide value is exposed simultaneously with a fresh sample of the same oil to light from CX tungsten lamps, it will continue to develop peroxides and at the same rate as does the fresh oil; (3) the induction period of an oil which has been protected by a sextant green filter is unaffected by the peroxides which were developed during said protection and is equal to that of a fresh sample of the same oil; (4) the development of rancidity in oils that have been protected by a sextant green filter, and which have subsequently been exposed to light, proceeds independently of the peroxides that may have formed during the period of protection; and (5) peroxides which develop under a sextant green filter do not add to the susceptibility of the oil to become rancid.

The Russell-effect procedure for obtaining pseudophotographs, by exposing a sensitized film over a rancid oil, has been standardized so that consistent results are now obtainable. Tests have indicated the possibility that hydrogen peroxide is the active substance which brings about rancidity and also causes the Russell effect, and that the organic peroxides have little, if any, effect.

### LOOSENING THE HULLS OF WALNUTS

It has been established that the hulls of sticktight California walnuts may be loosened by treatment with ethylene mixed with air in the ratio of 1 to 1,000 volumes for 60 hours if the nuts are sufficiently mature. Since there are appreciable losses of color of the kernels during the treatment, it is better to use temperatures up to 90° F. and finish in 60 hours than to use lower temperatures and longer time.

Owing to hot weather, the 1936 walnut crop was inferior in quality. Field sorting was recommended, in order to avoid putting sunburned nuts through the ethylene process. This practice, in conjunction with the ethylene treatment, raised the price received by growers in the interior districts about 4 cents a pound in a year when nuts were of less than average quality.

### ENZYME INVESTIGATIONS

The isolation of the papainlike proteinase which is present in wheat flour already has been reported. This enzyme has an unfavorable effect in the process of commercial breadmaking from normal flours, but it is of advantage when abnormally hard flours are used. Its protein-digesting action is inhibited by the common bleaching agents used in the manufacture of flour. During the year, further attention was given to the activators of this enzyme. It was found that minute traces of cysteine or glutathione were sufficient to produce a desirable softening effect on dough made from hard flour. Cysteine, in quantities of 0.1 to 0.5 percent, disperses wheat gluten completely. The product is a stable protein solution that looks like milk. The properties of this protein preparation are so curious that it was studied carefully and the results published. The dispersion of the protein is believed to be due to a chemical change, namely, the reduction of the sulphide groups in gluten. Practical use of the liquefied protein as an adhesive or as the base for a plastic material seems to be possible. Since the protein coagulates in the presence of electrolytes, its use in the fining of wines and other clarification processes is being investigated.

A study was made of the occurrence of fat-splitting enzymes in flour, because of their obvious connection with deterioration of flour in storage, such as increased acidity or rancidity. It was found that flour contains little or no enzyme material capable of hydrolyzing the ordinary fats. It does, however, contain an enzyme which easily splits the glyceryl esters of some of the lower fatty acids. Any lipolytic activity in flour must, therefore, lead largely to the production of volatile fatty acids. It was observed that, on the addition of the proper substrates, these volatile acids are produced very rapidly in the cold (just above freezing) as well as at higher temperatures. Cold storage of flour cannot prevent this type of deterioration.

The Food Research Division collaborated with the Hawaii Agricultural Experiment Station in a study of methods for extracting papain from papaya fruits and plants and bromelin from low-grade pineapple juice.

### EGGS AND EGG PRODUCTS

During the year a large-scale egg-oiling experiment was started at Omaha, Nebr., by a cold-storage company, in which 800 cases of eggs were divided and treated by three representative methods of oiling, including that developed by the Bureau. One-half of each lot was then shipped to New York, to determine the effect of transportation on the preservation of quality; the other half remained at Omaha in cold storage. It is estimated that 35,000 cases of eggs were treated by the Bureau's improved oiling process in 1936. A machine for applying this process commercially is being constructed by a private concern.

The Bureau's enzymic process for preparing egg whites for drying has been further developed. Three public-service patents (U. S. Nos. 2,054,213, 2,062,387, and 2,073,411) have been granted. Experiments were made to determine the value of proteolytic enzymes, other than trypsin, as thinning agents for egg white. Pepsin was used successfully to recover egg white from the foam, formerly wasted, which forms on the surface. There was some indication that papain could be substituted for trypsin. Incidentally, papain was found to have some unusual properties, notably a peculiar behavior in clotting milk. Even with excess activator present, milk appears to have the ability to render some of the enzymes inactive.

Attention is being given to methods for removing carbohydrate compounds, particularly glucosamine, which are present in dried egg white prepared by the Bureau's process and cause gradual darkening of the product, if it is not kept cool.

#### DRESSED POULTRY

In order to determine what chemical changes take place in the tissues of frozen poultry during storage, many chemical analyses were made on frozen chickens which had been stored for 3 years, and compared with results obtained on freshly killed poultry. With the exception of an increase in muscle glucose and in acidity of the fat, no marked changes resulting from storage were indicated by the data. Undrawn birds showed a greater increase in acidity of fat, whereas it appeared that increase in muscle glucose was greater in drawn birds. The development of acidity in the fat, due to the action of lipase, increases with time of storage and appears to be less influenced by differences in temperature than by prior treatment of the birds. The development of muscle glucose, presumably due to hydrolysis of glycogen, appears to be greater at the higher temperatures of storage.

#### ENZYME ACTION AT LOW TEMPERATURES

Fundamental research on the chemistry of enzymes and of enzyme action at low temperatures was carried on during the year as a project under the Bankhead-Jones Act of June 29, 1935, providing for basic research in agriculture. Five pure enzymes of the proteinase class were prepared, and their action on known proteins (casein and crystalline egg albumin) at low temperatures was studied. The enzymes, themselves, were found to be uninjured when frozen in solution and kept at  $-183^{\circ}$  C. for 2 days. The activity of these enzymes on proteins followed approximately the usual course of chemical reactions at lower temperatures until the medium became solid. The change of the system from liquid to solid reduced the activity of the enzyme at once to about one-half. Glycerin, which was added to the enzyme substrate to keep it liquid and allow observations below the freezing point of water, appeared to inhibit proteinase activity to a much higher degree at low than at high temperatures, which indicates that a preservative which is useless at high temperatures may be quite efficient in cold storage.

Low temperatures had a greater effect on the clotting action of enzymes than on the well-recognized protein-splitting action. Clotting is inhibited to such a degree by low temperatures that there seems to be little likelihood of any such action occurring under cold-storage conditions.

Protein splitting is definite but extremely slow at low temperatures. The proteinases are definitely not responsible for all the changes in the quality of frozen meats, because it was found that the activities of fat-splitting enzymes are less reduced by low temperatures than are those of protein-digesting enzymes.

The effect of temperature on changes in the digestibility of fats was studied in detail. At high temperatures the velocity of hydrolysis of saturated triglycerides varies directly with the number of carbon atoms in the fatty acid, reaching a maximum at 8 or 9 carbon atoms. At low temperatures the rate of splitting is greatest for fats of the lower acids in the saturated series and for fats of the unsaturated acids, such as oleic. The rapidity with which cream and butter deteriorate in storage obviously is due to the presence of fats of the lower acids, such as tributyrin. The hydrolysis of this fat at low temperatures was found to occur with unusual rapidity.

In order to gain information which will be more applicable to natural fats, research is being undertaken on the behavior of mixed glycerides toward fat-splitting enzymes at low temperatures. This will require a great amount of work in synthetic organic chemistry for the preparation of samples.

The results of the work on the action of enzymes at low temperatures are given in three papers which have been prepared for publication.

#### WAXLIKE COATINGS OF FRUITS

Research was continued during the year on the chemical composition of waxlike plant products, particularly those on the surface of fruits. It has long been known that the surface coating of fruits retards evaporation of water, regulates the exchange of gases between the internal and external atmospheres, and provides, so long as it remains intact, protection against invasion by bac-

teria, fungi, and, to some extent, insects. More recently it has been learned that the waxy substances of the cuticle are of prime importance in the application and subsequent removal of insecticidal sprays. Much effort has been expended by research workers and in commercial developments toward devising methods of making sprays spread over and adhere to the waxy surface of fruits, and of removing from the harvested fruit spray residues which have been encompassed by further deposits of natural wax. Since these waxy constituents occur in many waste products from the canning and preserving industry, a knowledge of their character and means for their recovery may lead to industrial uses, some of which have already been suggested.

During the year investigations were completed on the nonvolatile waxy residue remaining after distillation of Florida grapefruit-peel oil. This material was found to contain solid fatty acids (dotriacontanoic, linolenic, linoleic, and oleic), a sapogenic ketone of high melting point, the hydrocarbons nonocosane and hentriacontane, a phytosterol, and umbelliferon. Laboratory work was also completed on the identification of constituents in the waxlike coating of the Bing cherry. Palmitic, stearic, linoleic, and oleic acids were isolated from the saponifiable, and nonocosane from the unsaponifiable, fraction of the wax. Glycerol, the sapogenin ursolic acid, and d-glycosidyl-sitosterol were also found in the cherry wax. The quantity of waxy material on Bing cherries is rather low, the petroleum ether and ethyl ether extracts being only 0.8 and 0.1 percent, respectively, of the dried skins. A comparison of these figures with the corresponding percentage yields from apple and pear cuticles indicates that herein lies the explanation for the less efficient protective surface coating of the cherry.

#### FRUIT SKINS

All fruits and vegetables are enclosed by a heterogeneous membrane designated by botanists as the cuticle. This cuticle, commonly called skin, plays an important role in the life of the plant during growth and continues to do so until the plant or any particular plant organ is disintegrated by decay or other destructive processes. If the cuticle is separated from adjacent tissues and then extracted with ether, it may be divided into two main parts, the ether-soluble fraction or waxlike coating, and the ether-insoluble part, which is often referred to as "cutin." Knowledge concerning the chemical nature of cutin is rather meager, but it is expected that investigations now under way will prove it to be an esterlike substance which, on saponification, yields a number of solid and semiliquid acids. Attempts are being made to identify these products, in order to have definite knowledge concerning the chemical structure of cutin. Apple cutin, after being subjected to preliminary treatment for the removal of foreign matter, with the exception of a small percentage of cellulose, was saponified with alcoholic potash, and an attempt was made to separate the products so formed. Two main fractions thus far have been obtained, one an oily substance and the other consisting of one or more solid acids. Ordinary methods have so far failed to separate the solid acids, but molecular distillation of the acid esters offers some promise of accomplishing their purification.

#### APPLE PIGMENTS

The development of color in apples is important, both to the grower and the consumer. Well-colored apples almost invariably command higher prices, and they are the least susceptible to storage scald. The maximum development of color is, moreover, a criterion of that particular stage of maturity at which the fruit will attain satisfactory flavor and aroma and also be most edible in other respects. Owing to the economic value which is placed on color in apples, attempts have been made to determine the factors which are responsible for and influence the production of color. Before there can be a more thorough understanding of the complex subject of pigmentation in apples, however, it is necessary to acquire knowledge of the chemical nature of the pigments involved and of the biochemical reactions responsible for the formation of the red, or anthocyanin, pigment and of its chromogenic precursor. The results of an investigation on the coloring matter of Grimes Golden, Jonathan, and Stayman Winesap apples have been published. The anthocyanin pigment of Jonathan and Stayman Winesap apples was identified as idaein, a galactoside of cyanidin. The yellow pigment of Grimes Golden and Jonathan apples, which is soluble in the cell sap, has been identified as a previously unreported galactoside of quercetin. It may safely be assumed that the red pigment occurs in all red apples, and that the yellow pigment occurs in both red and yellow fruit. As



previously observed with the pigments of corn husks, the anthocyanin, or red, pigment is the reduction product of the yellow pigment. Red apples obviously possess the complete mechanism for converting the quercetin galactoside or chromogen into red idaein. In apples of the Grimes Golden type apparently there is lacking or inhibited some factor other than the chromogenic substance, since the latter is present but not capable of being reduced to its homologous anthocyanin.

#### PLANT VIRUSES

In cooperation with the Bureau of Plant Industry on a Bankhead-Jones project for the study of plant viruses, the Bureau is making studies on their character and properties.

Quantities of the crystalline protein known as the virus of green tobacco mosaic were prepared by the method recently published by W. M. Stanley. It was found possible to shorten this procedure and produce crystals which were as infectious as those prepared by the longer method from comparatively crude tobacco protein after digestion with trypsin.

On several occasions modifications of Stanley's method resulted in the production of protein crystals which differed decidedly from the ordinary form. Such atypical crystals were found to be infectious, but their real significance and importance has not yet been determined. It is probable that they represent merely a second crystal form of the same substance.

Further work on the tobacco mosaic consisted of a comparison of the enzyme systems of normal and infected plants.

In experiments on the respiratory rate and respiratory quotient of mosaic and healthy tobacco tissue, it was found that the respiratory rate varied widely with the age of leaf chosen. No significant differences could be established between comparable leaves of healthy and diseased plants.

From studies of the amylase content of tobacco leaves, not yet completed, it appears that infection with yellow mosaic results in an increase in alpha amylase, whereas infection with green mosaic does not.

Efforts are being made to find a clue to the action of the virus in the plant and a means for measuring the potency of the virus by a chemical determination rather than by a plant assay.

#### MINERAL CONSTITUENTS OF PLANTS

Toward the close of the year, work was started on a new project which has for its object the collection of all available information relating to the effects of soil mineral constituents on the mineral content of food plants and the effects of plant mineral constituents on animal and human nutrition. This work is being done in connection with the Department's program of basic research in agriculture, provided for in the Bankhead-Jones Act of June 29, 1935, to determine the need for possible cooperative research on this subject. It involves a thorough search of chemical literature and also the collection of the very extensive unpublished results of the State agricultural experiment stations which are giving their cooperative assistance through careful attention to a questionnaire.

#### TOXICITY OF INSECTICIDES TO HIGHER ANIMALS

During the year extensive building alterations were made at Stanford University, Calif., in order to provide adequate space and facilities for the Bureau's cooperative pharmacological studies on foods. These studies are concerned with the acute and chronic, but chiefly the chronic, intoxications which may result from the consumption of food and food products contaminated with insecticidal-spray residues, food preservatives, and metals which may occur naturally in foods or as the result of canning and cooking processes.

Investigations were made on the isolation and identification of the compounds excreted in the urine, and on the relationship of hydrogen-ion concentration, oxygen consumption, and the bactericidal properties of the urine, following the oral administration of phenothiazine. Toxicity studies on methyl and lauryl thiocyanate and on anabasine have been completed. The toxicity of thiocoumarin, paranitrobenzene, derris, and pyrethrum also received attention. Experiments were made on the production of anemia in rats by feeding diets containing fluorine, in the form of cryolites, and on the production of exostosis of the bones in rabbits by repeated injections of sodium fluoride.

Experiments on the continued feeding of dicalcium phosphate containing from 0.25 to 0.27 percent of fluorine to albino rats showed that the presence of large

quantities of calcium and phosphoric acid does not reduce or antagonize the toxicity of fluorine. The toxicity of fluorine, present as a contaminant in dicalcium phosphate, is as great as that of sodium fluoride.

Three papers were published as a result of the studies on chronic toxicity of nicotine. The chronic effects of nicotine, resulting from continued feeding of nicotine-containing diets, can be accounted for very largely by the decreased food intake, which of itself would cause stunted growth. Data have been obtained on the organ weights of rats used in the experiments, but the significance of changes in organ weights cannot be evaluated until data are accumulated showing the relation of organ weights to food intake. Experiments to supply these data have been started.

As in the case of nicotine, the changes in growth rates, resulting from continued feeding of anabasine, can be largely accounted for by the lowered food intake.

## INDUSTRIAL FARM PRODUCTS RESEARCH

The Industrial Farm Products Research Division concerns itself with the utilization of agricultural products for nonfood purposes from three angles, namely, (1) improvements in the raw materials and in processes now employing agricultural products, (2) new and more extended uses for agricultural products already employed in industry, and (3) new uses for agricultural products now considered useless for industrial purposes. The following statements regarding work in the various sections of the division are intended to show what progress has been made in these directions during the year.

### HIDES AND SKINS

The results of extensive observations in an almost virgin field of research, namely, the influence of animal feeding on the leathermaking characteristics of the skins, have been assembled and approved for publication. These studies yielded data of special value because of their fundamental and quantitative correlation of life factors with the characteristics of the animal skin. Nine sets of twin lambs were used in the feeding experiments. The twins were divided into two groups, one of which was full-fed and the other underfed. The final average body weight of the underfed lambs was 56 percent of the weight of their full-fed twin brothers. Based on natural thickness, leather from the skins from the underfed group had a tear resistance equal to 54 percent of that of the leather from the full-fed animals, a tensile strength equal to 60 percent, and stretch at break load equal to 81 percent. The skins and leather from the full-fed animals had distinctly larger collagen fibers with a high angle of weave, a more open fiber structure, and a much higher grain, the latter being dependent on the relative development of skin fibers and wool fibers during growth.

The results of these studies on lambskins, showing the influence of feeding, have aroused keen interest in the trade among both the scientific men and the tanners. This is especially true of the tanners of calfskins, who have urged that similar studies be undertaken with calves at the first opportunity. Toward this end and for fostering a broader and more effective program of research on the influence of animal-life factors on the leathermaking characteristics and value of hides and skins, a formal cooperative agreement has been entered into with the Bureau of Animal Industry.

The observations and data obtained from previously described experiments on the curing of calfskins with salt plus small quantities of special chemicals have been published. Aside from showing specifically the effectiveness of the chemicals used, this work has demonstrated the economic possibilities in developing methods of curing that will stop the action of bacteria and molds on hides and skins. In a continuation of these studies, other chemicals mixed with salt have been tried out on a small scale, but so far no treatment has been found that appears more promising than the mixture of salt, sodium silicofluoride, and paranitrophenol used in previous experiments.

Small-scale laboratory experiments have been made on the effectiveness of various denaturants mixed with salt for the resalting of hides and skins. Since only 3 or 4 pounds of very fine salt are sufficient to obtain a uniform distribution of salt over the flesh side of about 60 pounds of salted calfskin, it was deemed necessary to increase the ratio of denaturant to salt over that found effective for salting green hides. A mixture containing sodium silicofluoride and paranitrophenol appeared distinctly the most effective of the four mixtures tried. All four, however, showed better preservation than with salt alone.

In connection with the addition of fluorides to salt for curing calfskins, consideration was given to the possibility of introducing fluorine into gelatin made from them. The fluorine content of commercial gelatins is from 4 to 10 parts per million. Experimental gelatins made in the laboratory from calfskins cured with salt alone were found to have about the same fluorine content. The fluorine content of gelatins made from calfskins cured with salt to which fluorides had been added was found to be materially influenced by the manner of washing the skin prior to lining. With thorough washing in running water these gelatins had about the same fluorine content as the gelatin from calfskins cured with salt alone, but when washed by still soaking a high fluorine content resulted. Impurities in commercial salt capable of forming difficultly soluble fluorides may markedly influence the fluorine content of gelatin made from calfskins cured with salt plus fluorides.

Both naphthalene and paradichlorobenzene have been used commercially for preserving salted hides and skins. Studies on the toxicity of these chemicals in the vapor phase to fungi isolated from salted hides indicated that the vapors of these chemicals are not fungicidal but do inhibit the growth of mold. It was found that fats, which are always present in hides and skins, will absorb enough of the vapors to materially prolong inhibition of the growth of mold.

Continuation of studies on the physiology of hide-reddening organisms has established that these organisms when producing red pigmentation cause an oxidative type of break-down in protein substrates. These same organisms, when grown on media of low salt content, do so by a reduction of protogenous substrates without the formation of reddening or pigmentation.

A long-time semicommercial experiment planned to observe the keeping quality of salted calfskins in cold storage over a period of at least 4 years is under way. Each skin was divided into two sides to be held in storage for different periods. One-half of the first lot of calfskin sides was tanned into finished leather a year ago. The second half of this same lot is now being tanned in the same tannery and by the same processes. The sides in the second half were 31 months old when put to soak and had been in cold storage 26 months. From all outward signs they appeared to be in excellent condition. When tanned, these sides will be mated with their respective opposite sides from the same skins, which were processed 1 year ago. The sides will be selected, graded, tested, analyzed, and otherwise directly compared with each other, for determination of the deterioration, if any, resulting from additional cold storing for 1 year. The tanning of one-half of the sides of the second lot has been started for comparison with mate sides to be taken from cold storage 1 year hence.

#### TANNING MATERIALS

The leather industry of the United States consumes annually about 115,000 tons of tannin, equivalent to 460,000 tons of 25-percent extract. It imports practically half the tannin it consumes. Two-thirds of its total consumption comes from two raw materials, namely, chestnut wood and quebracho wood.

Tannins are used to make vegetable-tanned leathers, especially heavy leathers, such as sole, belting, harness, case, bag, and strap.

One of the most comprehensive surveys and studies ever made of a tanning material has been published in Department of Agriculture Technical Bulletin No. 566, entitled "Western Hemlock Bark an Important Potential Tanning Material." This study was made to gather and present facts on which those who might be interested could base a decision as to the feasibility of salvaging waste hemlock bark of the Pacific coast region in the form of tanning extracts for making leather.

It is shown that western hemlock bark yields a high-grade extract capable of making satisfactory leather when used alone or blended with other tanning materials. About 5.5 tons of the bark will yield 1 ton of powdered extract containing about 55 percent of tannin and having a market value ranging from \$88 to \$110 a ton. This would give a value for the waste bark ranging from \$5.50 to \$8 a ton. It is probable that the extract, after it has been introduced, would bring about 8 cents a tannin unit in the eastern markets. Until the merits of the extract have been established, however, this price will meet with serious sales resistance.

It is estimated that, with present operations, in Washington and Oregon there are available annually for making into tanning extract at least 50,000 tons of western hemlock bark. By modification of logging practices, the supply

probably could be increased to 200,000 tons of bark having a potential value in the form of tanning extracts of over \$3,000,000 annually.

A formal relationship has been established with the Bureau of Plant Industry for a program of research covering tannin-bearing plants and their development, preferably in the form of new crops. One of the first steps, which is now under way, is a review and analysis of available information to select those plants offering the most promise of successful development.

Some interesting data on tannin content have been obtained from a special collection of samples of *Lespedeza sericea* from the Bureau of Plant Industry. *L. sericea* is an Asiatic legume introduced into this country as a hay-grazing crop, especially for the Southeastern States. The collection comprised 20 samples of separated leaves and stems cut at weekly intervals from May 29 to July 31, 1935, from experimental plots at the Arlington (Va.) Experiment Farm. The tannin content of the leaves, by the hide-powder method of the American Leather Chemists' Association, increased steadily from 7.5 percent in the youngest cutting to 17.1 percent in the oldest. Most of the tannin is in the leaf of the plant, the tannin content of stems being just a little over 1 percent. On the basis of these results, *lespedeza* should be cut for hay as early as possible. Even then the tannin content may be high and in later cuttings may be great enough to seriously interfere with assimilation by livestock.

#### LEATHER

Strictly comparable studies with laboratory-tanned leathers have shown that incorporation within vegetable-tanned leather of sodium chloride or sodium tartrate, 2.5 and 5 percent, respectively, imparts high resistance to acid rot. Although both salts are very effective, sodium chloride is preferable because it is cheap, practically neutral, and needs no adjusting of the pH value of its solutions when applied. Furthermore, sodium chloride, unlike alkali salts of weak organic acids, does not increase the absorption by the leather of acids from the atmosphere. In equal quantities it affords greater protection than sodium tartrate and would appear to do so over a longer period of time.

The results of these studies, which have been published, have raised an especially fundamental question as to why and how sodium chloride acts as a protective agent in retarding the acid rot or red rot of leather. As a result of this work two tanners have reported that they are now putting sodium chloride into bookbinding, upholstery, and similar leathers.

In cooperative studies with the Government Printing Office on bookbinding leathers, a chrome-tanned sheepskin, the first leather of this type ever purchased by the Printing Office for bindery work, was tested for permanence. On exposure to accelerated rotting in the gas chamber this leather lost, after 12 weeks, only 11 percent of its strength, and after 18 weeks, 16 percent, indicating exceptional permanence. This is especially noteworthy, as the leather was made from sheepskin which ordinarily has a low rating for permanence. These results suggest a means of utilizing sheepskin for binding and similar purposes, where permanence is a dominating element. Tanners of sheepskins should recognize this possibility and should show a more progressive interest in it.

Previous work has shown that combination vegetable-chrome retanned leathers are more permanent than straight vegetable-tanned leathers. Data that have been acquired on commercially made combination leathers, however, show that all leathers of this general tannage do not have essentially the same resistance to decay. In fact, some of them show but little more resistance than straight vegetable-tanned leathers. Apparently there are unknown factors involved in the making of leathers of this character that exert a pronounced influence on their performance, one of which may be the chromic oxide content. Consequently, an elaborate set of laboratory-tanned combination leathers and straight vegetable-tanned leathers was made. In this set the combination-tanned leathers included a range in chromic oxide content from about 0.5 to 3 percent. These leathers have been exposed to accelerated aging, tested, and analyzed. The data are being assembled for publication. Although definite conclusions are as yet premature, the indications are that the resistance to decay of the combination vegetable-chrome tanned leathers is not proportionate to the chromic oxide content.

For a number of years the research work by this Bureau on the causes and prevention of the rotting of leather by acids absorbed from the air has been promoted by the use of a gas chamber for accelerated rotting. The value of such an apparatus in obtaining results within several months rather than after

many years has now been definitely demonstrated. As a result a number of tannery chemists have voiced the desirability and usefulness of a standardized gas chamber, not only for the study of the effect of changes in their own particular processes, but also for comparative work between different laboratories. The present gas chamber depends for its corrosive atmosphere on the burning of city illuminating gas. This obviously varies greatly throughout the country. In response to the expressed need of such equipment, much attention has been given to the development of a duplicable gas chamber. Decided progress has been made in designing and building an apparatus of this type. When it is completed, data on its performance will be acquired for publication, together with a description of the chamber.

Through committee memberships cooperation has been continued with the American Leather Chemists' Association in research work on leather, and with the Federal Specifications Executive Committee in the drafting and revising of specifications for leather and leather goods to meet the requirements of the Federal Government.

#### FARM FABRICS

During the year experiments were continued to determine the effectiveness of copper compounds as mildew-proofing agents for cotton fabrics, particularly when used as constituents of mixtures for the mineral dyeing of cotton duck. A manuscript giving the results of this investigation has been prepared for publication, which will close the project.

#### UTILIZATION OF FARM WASTES

In the Agricultural Byproducts Laboratory at Ames, Iowa, small-scale destructive distillations were made on almond shells, bagasse, flax shives, rice hulls, grape pomace, olive pits, and date seeds, using a closely controlled electrically heated retort. Analyses of the destructive-distillation products from these materials were completed and the data compiled. The results of this work are being correlated and assembled in a general report on the destructive distillation of farm wastes.

A new cellulose laboratory has been established and equipped with the necessary experimental apparatus for small-scale pulping and bleaching, alpha-cellulose production, and viscosity work on cellulose. A constant-temperature humidity room has been built for conditioning pulp and paper samples before testing. Samples of commercial pulps have been collected from manufacturers as standards of comparison in the production of paper pulp from the cellulose fibers in crop residues. Investigation is under way to ascertain the effect on pulping action of adding inorganic salts to pulping liquors. Coordination of bleaching methods has been started, both at Ames and in Washington.

The practicability of nitric acid as a pulping agent for sugarcane bagasse or other residues depends on reuse of the liquor. Fortunately, used nitric acid liquor has been found to be a better pulping agent than pure nitric acid. Investigations have shown that acetic, formic, and oxalic acids are formed when dilute nitric acid acts on lignocellulose material, and these have a beneficial effect. Preliminary work has been started in glass with alcoholic nitric acid with promising results. Small-scale nitric acid pulping experiments have been made on both straw and bagasse. Equipment made of special acid-resistant alloy has been designed and ordered for pilot-plant-scale experiments.

The success of any pulping process depends on efficient utilization of pulping agent and the recovery of chemicals from the spent liquor. This is especially true in the pulping of bulky crop residues, due to the large volume of dilute solution necessary to cover the material and the large quantity of chemicals required. For the purpose of determining the degree of consumption of chemicals in the pulping process, a cheap, compact, and accurate instrument has been devised, which quickly gives direct readings of electrical conductivity of solutions by adjusting a slide-wire rheostat. This instrument may be used with the ordinary 110-volt, 60-cycle, alternating current. With its aid the different degrees of "spentness" of caustic soda solutions, or the consumption of caustic in the formation of sodium carbonate and organic sodium salts, may be closely followed. It has been used for determining the direct-causticizing effect when lime is added to spent liquor. The results, which are being prepared for publication, show that the direct-causticizing treatment is effective and practical.

## LIGNIN

The lignin compounds or complexes obtained as byproducts of pulp making have proved useful for treating natural water supplies to remove iron and manganese and to reduce the quantity of fluorine present. They compare very favorably with commercial products sold for these purposes. An application for a public-service patent covering this use of lignin complexes has been filed. Treatment of water containing carbon dioxide with lignin and some lignin complexes releases the gas from solution so that it can be removed easily. Removal of carbon dioxide is very desirable in the instance of boiler feed waters, and lignin may eventually be used in large volume for this purpose.

The results of the study on ammoniation of waste sulphite liquor for fertilizer use, previously reported, have been published.

A study was made on the chemistry of lignin from oat straw and rice straw. In connection with this work it was necessary to develop a new analytical procedure for determining percentages of methoxyl and ethoxyl when both of these groups are present.

The nature of the complicated chemical reactions by which lignin is synthesized in the living plant is also under investigation. The oat plant was selected for this study, samples being taken at weekly intervals from the time the seedlings emerged from the ground until maturity.

Some work has been done on the dehydrogenation of lignin from corncobs with selenium and on the chlorination of lignin.

## FERMENTATION PRODUCTS

Experiments were made with cornstalks, in order to determine the break-down into gaseous products caused by the bacterial flora which may be present in the material. Various controlled temperatures and periods of time were used. Complete analytical data on starting material and products of reaction have been compiled. Similar experiments were made with wheat straw, flax straw, Jerusalem-artichoke tops, and spent cook liquor from the manufacture of wallboard. It was found that the chemical composition of the material, as indicated by determinations of lignin, cellulose, and pentosans, was not a dependable criterion for predicting the results of anaerobic fermentation. Apparently physical factors play an important role. A report on the effect of temperature of digestion on the production of fuel gas from farm wastes has been submitted for publication.

For pilot-plant-scale experiments on the production of organic acids by mold fermentation of corn sugar, a 400-gallon aluminum rotary fermenter, together with accessory equipment, was installed in the Agricultural Byproducts Laboratory at Ames, Iowa. Experimental work with this equipment has demonstrated that the mold fermentation processes previously developed in the laboratory can be carried out on the larger scale without loss in efficiency or ease of operation. This equipment has aroused a great deal of interest, and a number of commercial firms have requested permission to make use of the principle underlying its design.

Up to the present, the large rotary fermenter has been used only in the manufacture of calcium gluconate, of which over 1,000 pounds were made from corn sugar. Results have indicated that calcium gluconate can be made more cheaply by this method than by any other known. Experiments with the small rotary fermenter have shown that it is possible to use this same type of equipment in the production of a pure dextrolactic acid from corn sugar and the oxidation of sorbitol to sorbose. Both processes will be tried in the semiplant-scale fermenter when the small-scale experimental work has been completed.

## POWER ALCOHOL

Preliminary to investigations on the production of power alcohol from agricultural products, a general survey has been made of the problem. This work was undertaken because of the increasing consumption of motor fuel with a consequent decrease in irreplaceable petroleum reserves, the existence of farm surpluses and wastes which might be processed for use as fuel, the popular interest and growing industrial development in the production of ethyl alcohol from saccharine and starchy plant products for specific use as motor fuel, the increasing interest in the possibility of producing farm crops specifically as raw materials for industry, and the need for sources of power for farm use, which could be produced on the farm.

The chief activities during the year have been the making of contacts with Government agencies, equipment manufacturers, and producers of products suitable for fuel; the collection of statistics on the production of fuels now used and of possible raw materials including crop surpluses, culls, and wastes; the study of possible processes and plant designs for the production of fuels from agricultural materials; the establishment of patent and literature files on the subject; the inspection of alcohol plants; and similar work necessary for a general survey of the problem. A report has been prepared summarizing tentative findings to date.

#### CHEMICAL CONVERSION OF OILS, FATS, AND WAXES

During the year the Bureau entered on a comprehensive program of research, the purpose of which is to convert agricultural oils, fats, and waxes into new products useful in industry. The ultimate products are expected to include plastics, wetting agents and other textile and leather-finishing assistants, pharmaceuticals, soaps, dyestuffs, germicides, lubricants, synthetic waxes, and rubberlike compounds. Preliminary work has been done on the preparation of amino derivatives of fatty acids which may find use in the compounding of rubber.

#### INDUSTRIAL UTILIZATION OF SOYBEANS

The Regional Soybean Industrial Products Laboratory was established toward the close of the fiscal year 1936 in quarters furnished by the University of Illinois at Urbana. Its purpose is to conduct research, in cooperation with the Bureau of Plant Industry and the agricultural experiment stations of the North Central States, on soybeans and soybean products designed to promote their industrial utilization. The work at this laboratory is part of the Department's program of basic research in agriculture, provided for in the Bankhead-Jones Act of June 29, 1935. It includes analyses of soybeans and soybean products as an aid to the agronomic and technological research, studies on the properties and industrial utilization of soybean-oil meal and soybean oil and their constituents, including proteins, carbohydrates, glycerides, fatty acids, phosphatides, sterols, and associated compounds, and studies of the engineering and economic aspects of the industrial utilization of soybeans and soybean products.

Active work in most of these lines has been in progress during the year, although there were some delays caused by the necessity for designing and procuring special equipment and of making preliminary studies.

Analyses have been completed on 166 samples of soybeans representing 9 different varieties. Thirteen separate determinations were made in the analysis of 77 of these samples, and 8 determinations were made on each of the others. Although the samples analyzed represent only a fraction of the total experimental plantings in 1936, some very interesting indications have been noted. All varieties grown at Ames, Iowa, gave a uniformly high yield of protein, and the oil possessed a high iodine number. On the other hand, varieties grown at Columbia, Mo., showed a definite trend toward the formation of oil having an abnormally low iodine number.

For studying the chemistry of the fatty acids and the composition of the glycerides of soybean oil, a sample of oil was prepared carefully from a bushel of beans of known variety and history. The usual constants have been determined, and the fatty acid constituents are now being determined in detail.

Crude sterol mixtures were separated from soybean-expeller oil and subjected to various purification processes to remove nonsterol components. Attempts to isolate and identify stigmasterol, which is of great value in the synthesis of sex hormones, have been unsuccessful thus far. A sterol glycoside has been isolated in pure form, but neither the sterol nor the sugar component has been definitely identified.

In connection with studies on the possibility of improving the stability of edible soybean oil, experiments were carried out which established the fact that trimethylamine oxide, a break-down product of lecithin, reacts with oleic, linoleic, and linolenic acid esters to yield various substituted amines and unidentified products and that the fatty acid esters, especially the linolenic ester, acquire odor characteristics which closely resemble those of a so-called badly reverted soybean oil.

Quick-drying varnishes of pleasing appearance were made from blown and heat-bodied soybean oils, in combination with various synthetic resins. Attempts were made to improve the drying quality of soybean oil for use in paints by treatment with various driers. Although some success was attained, a definitely

satisfactory drier for use in soybean oil paints has not been found. Panels coated with soybean varnishes and paints have been prepared and placed on racks for weather-exposure tests.

Some preliminary work has been done on the preparation of paper coatings from protein material extracted from soybean-oil meal by water and by weak alkali. These protein extracts have been found to vary in dispersibility and viscosity, and possible causes of these variations are being investigated.

#### CHEMICAL WEED KILLERS

In collaboration with the Bureau of Plant Industry, a semi-industrial scale electrolytic sodium chlorate plant was successfully operated, and about 7,000 pounds of this herbicide were made and shipped to agricultural experiment stations, designated by the Bureau of Plant Industry, for use in field studies on the control of noxious weeds. From economic studies it was concluded that sodium chlorate can be produced at 4.9 cents a pound. This figure excludes sales and promotion costs and corporation taxes, but includes 5 percent interest on investment. At present, the base selling price in carlots from the sole American producer is 6.25 cents a pound. The farmer pays from 9 to 10 cents a pound for small purchases. A report on the cost of manufacturing sodium chlorate, based on this investigation, has been submitted to the House Appropriations Committee of Congress and will be published in abbreviated form.

In collaboration with the Bureau of Plant Industry, a new herbicide has been developed. It comprises nitric acid mixed with a dehydrating mineral acid, such as phosphoric or sulphuric acid. The presence of oxides of nitrogen or copper salts in these acids appears to be advantageous.

#### NAVAL STORES RESEARCH

##### CHEMISTRY OF NAVAL STORES (TURPENTINE AND ROSIN)

In connection with experiments on the dehydrogenation of fractionated rosin products from pine gum it was noted that palladium carbon, normally a dehydrogenating catalyst, also brought about an isomerization of the resin acids and that the resulting products were essentially pyroabietic acids which are comparable in stability with devtropicmaric acid, one of the most stable of the known resin acids. The high stability of pyroabietic acids, both as regards isomerization and oxidation, renders them superior to abietic acid or any of the mixed acids present in rosin. The only means used heretofore for the preparation of pyroabietic acids involved heat treatment of rosin or resin acids which resulted in a complex mixture, from which the separation of the pyroabietic acids was exceedingly difficult.

A rapid method for the isomerization of rosin and resin acids by catalytic means was devised. The catalytic procedure accomplishes the isomerization within a period ranging from 15 to 30 minutes, in comparison with from 80 to 100 hours required for the ordinary heat treatment, and produces a much better yield of the desired pyroabietic acid. Several catalysts were found to be effective, but palladium carbon was the best.

A number of resin acids and rosins from various sources were found to yield alphapyroabietic acid almost exclusively by the catalytic process of isomerization. The production of alphapyroabietic acid from resin acids and rosins from different sources by catalysis is of scientific, as well as industrial, interest because previous records indicate that resin acids and rosin from different sources yield different pyroabietic acids.

In the investigation of the components and derivatives of pine oleoresin, attention was directed during the year to the so-called neutral substances in the nonvolatile portion. These neutral substances, although present only to the extent of about 5 percent, have an influence on the properties of the rosin made from pine gum. An efficient automatic laboratory process for separating this group of substances from comparatively large quantities of gum was worked out. The neutrals from longleaf pine gum were found to be a complex mixture consisting of difficultly saponifiable esters of the abietic type, polymerization products, and ill-defined unsaponifiable oxygenated bodies similar to and including the so-called resenes. Certain components, which were found to be somewhat volatile under a high vacuum, closely resembled some of the less volatile components of the "tailings" left in the fractional distillation of



gum spirits of turpentine, and it is expected that further study of these components will throw light on the less-known tailings constituents of gum spirits.

Progress was made in the development of special methods of saponifying esters of the abietic type by conducting the saponification at high temperatures in solvents having high boiling points.

A study was made of methods for determining the degree of unsaturation of rosin products. A high degree of unsaturation is indicative of instability. Stability is a particularly important property in rosins from the point of view of industrial usefulness.

A laboratory technique, which does not require the use of fractionating columns, has been worked out for the identification of steam-distilled wood turpentine and differentiating it from gum spirits of turpentine. It involves the isolation of benzaldehyde and fenchyl alcohol and their subsequent identification in the form of characteristic derivatives.

A process for chlorinating turpentine was devised, in which the hydrochloric acid which is evolved, when chlorine is substituted in the pinene molecule, is utilized in the preliminary steps. By first saturating turpentine with hydrochloric acid and then with chlorine, a final product was obtained which contained more chlorine and was more stable to heat and light than when chlorine alone was used.

When rosins are dissolved in solvents for the preparation of gloss oils, spirit varnishes, and adhesives, crystals sometimes separate on standing. This makes the rosin solution unsuitable for the purpose for which it was intended. During the year a simple method was tried out for determining the tendency of any particular lot of rosin to crystallize from solution. This method consists of placing the rosin in small lumps in a test tube, covering with the solvent, and allowing to stand. A solution is formed, which varies from saturated at the bottom to very dilute at the top, and at some point the concentration is optimum for inducing crystallization. It was found that crystallization occurred much more readily by this method than when a uniform solution of 1 g of rosin per cubic centimeter of solvent was used. Fifteen samples of gum rosin, which gave trouble in commercial utilization because of crystallization, were found to crystallize from alcohol solution overnight, whereas most samples of gum rosin did not form crystals on standing for 2 days. Acetone proved more effective than alcohol for inducing rapid crystallization.

The quantity of metals in gum rosin is remarkably small, but the presence or absence of certain metals may be of importance in connection with certain uses of rosin. The ash of most rosins contains enough iron and zinc, or aluminum, to be identified by chemical means. Copper can be detected only occasionally, unless very large samples are used. Last year, with the collaboration of the Fertilizer Research Division of this Bureau, the presence of various metals in the ash from rosin was determined by spectroanalysis. A spectrogram of the ash from 100 g of commercial gum rosin of WG grade showed the presence of the following metals in the order of their abundance: Zinc, iron, lead, barium, aluminum, copper, manganese, magnesium, silicon, and boron. Similar determinations are being made on the ash from pine gum itself.

Efforts were made to find new uses for the compounds of resin acids with various metals. A copper resinate spray solution containing copper equivalent to that in a 5-5-50 bordeaux mixture was tried out for preventing late blight of potatoes, in cooperation with the Maine Agricultural Experiment Station, and was reported to give effective control under severe blight conditions. A dusting powder rather than a spray is desired, however, and work toward this end is being continued. Silver, cadmium, and copper resinates were tried out as mildew-proofing agents for cotton fabrics and found to be effective as long as they remained in the fabric. Weathering removes them in about 6 months. From experiments in which these resinates were used in combination with beeswax, however, it appears that their incorporation with wax finishes used commercially for waterproofing canvas would be feasible, and that they would not be readily removed when thus incorporated.

Processed oleoresin is now a commercial product and is being used experimentally by some industries. Advice has been given with regard to methods for determining the percentage of spirits of turpentine in the oleoresin and methods of distillation to insure recovery of all the turpentine without overheating and without having entrained rosin in the turpentine.

Examination of 10 samples of steam-distilled wood turpentine and 4 samples of destructively distilled wood turpentine, which had been stored for 9 years in large glass bottles in a moderately cool place and exposed only to subdued

daylight, showed that in instances where the bottles were full and tightly stoppered there had been no material changes in color or specific gravity.

Under the usual storage conditions, turpentine undergoes changes in composition, viscosity, and specific gravity due to oxidation, polymerization, and other action and becomes discolored from contact with metals, particularly iron and copper. It appears that at least some of the undesirable changes are promoted by the presence of dissolved or free water in the turpentine. Observations on samples of dehydrated turpentine, turpentine saturated with water, and turpentine containing free water, which had been stored for 3 years in contact with iron or zinc, and in some instances with oxalic acid crystals, showed that the turpentine in contact with water and metal did not change appreciably in specific gravity, although there was a decided darkening of color (most of which occurred during the first year) caused by the iron. The turpentine which had been in contact with zinc and water was still water-colored. Oxalic acid was not effective in preventing discoloration of turpentine by iron over the full period of storage, although it did prevent discoloration during the first year. The sample of turpentine used as a control had not changed appreciably in color, but it increased by 0.0015 in specific gravity. The turpentine which had been dehydrated with calcium chloride darkened two shades in color and increased by 0.0108 in specific gravity.

#### TECHNOLOGY OF NAVAL STORES

Experimental and demonstration work on the processing of pine gum was continued at the naval stores station in the Osceola National Forest at Olustee, Fla. The purpose of this work is to improve equipment and technique and thus lower the cost of production, yield standardized products, and prevent deterioration and losses in the handling of gum and its products.

A joint study of the fire still by the naval stores station and by cooperative agents in the field resulted in further changes in the standardization of fire-still plants. Plans and specifications were prepared for four types of plants adapted to different kinds of location and scale of operation. Studies on the distribution of heat in the fire path around the kettle led to changes in relative sizes of flues, whereby more even distribution of heat is obtained.

Approximately 550 barrels of gum were distilled with the fire still, principally for demonstration purposes. On the average, 14.2 man-hours were required for a 10-barrel charge. This is slightly more than the requirement for distilling crude gum with the steam still. The fuel requirements were less for the fire still than for the steam still.

An improved method was developed for distilling scrape in the fire still. It involves the addition of 1 barrel of water and 1 barrel of turpentine per charge and distillation in the usual manner. This method prevents the scrape from scorching, and thereby lowering the grade of rosin.

The steam turpentine still was used for the distillation of 1,500 barrels of cleaned gum and scrape diluted with from 9 to 43 percent of additional turpentine. On the average, 9.5 man-hours were required for a 10-barrel charge. In previous work with crude gum 14 man-hours were required. The time of charging the steam still with cleaned gum was reduced from a minimum of 30 minutes to 6 minutes by replacing the pump with an aluminum blow tank which also eliminates loss of turpentine in transferring the hot gum from the settling tank to the still.

The steam still, run intermittently as it is at the naval stores station, does not show any great advantage over the fire still in yields or costs. It is believed, however, that it would show greater efficiency if run continuously by large gum buyers or in connection with naval stores operations of 50 crops or more.

The gum-cleaning process previously developed was used experimentally in cleaning about 1,800 barrels of gum. Briefly, this process consists in filtering hot gum under steam pressure, subsequently passing turpentine through the equipment to clean the chips and filter medium and dissolve scrape, adding the turpentine to the filtered gum, adding hot water to and mixing it thoroughly with the gum and turpentine, allowing to stand overnight, and drawing off the lower layer of wash water which contains soluble impurities.

The investigational work on gum cleaning has yielded the following results thus far: (1) Development of a simple process involving filtration and washing; (2) production of a clean rosin having an average brightness of 96.2 percent, compared to 80 percent or less for commercial rosin from uncleaned gum; (3) production of rosin which is usually one or more grades lighter in color than

rosin made from the same gum uncleaned; and (4) substantial increase (from 2 percent for straight gum up to 8 percent for straight scrape) in the yield of rosin due to elimination of rosin losses in chips and dross.

Work was continued during the 1936-37 naval stores season with the turpentine-gum cups which had been under test during the previous three seasons, namely, galvanized iron, zinc, aluminum, clay, lead-coated galvanized iron, and gumwood. Gum collected in the galvanized-iron cups continued to yield low grades of rosin (D to H), and the cups were discarded at the end of the season; gum collected in the lead-coated and gumwood cups yielded medium grades of rosin (I to N); and that collected in the zinc, aluminum, and clay cups yielded pale grades (M to WW).

Experiments with cone-shaped wood cups, provided by the Forest Products Laboratory, gave promising results. Such cups can be made from the cores left from the manufacture of veneer, which now are used only for fuel. Molded cups made from phenol-formaldehyde synthetic resin were tried also, but these imparted a yellow color to the gum, which darkened the rosin. Continued experiments with cone-shaped glass cups indicated that these offer great promise.

In preliminary tests on the use of hydrochloric acid to stimulate the flow of pine gum, the application of acid to a normal streak (one-half inch) caused a gradual decrease in the yield of gum. The application of acid to a 1-inch streak increased the yield by about 50 percent.

Continued experiments on the comparative serviceability of galvanized-steel and white-oak dip barrels indicated that the metal barrels have certain advantages, as follows: They do not leak, except at top head; they have a definite and constant capacity; they have constant tare weight; they last longer than wood; and there is little or no cost for keeping them in good condition. The disadvantages of the metal barrels are that they are not obtainable locally; their first cost is higher than that of wooden barrels; they are harder to handle; they are easily dented; and the galvanized coating breaks down slowly, allowing the gum to be stained by iron.

In experiments on the straining of rosin it was found that, by increasing the "turning-out" temperature from 315° to 325° F., better and more rapid straining was obtained without apparent injury to the rosin. It was also found that straining will be satisfactory if the charge is turned out when the temperature in the still is rising instead of falling. Tests with different kinds of strainer wire indicated that 30-mesh bright steel wire produces as good a strainer as more expensive copper or brass wire of similar mesh, and furthermore it will not stain the rosin.

Seasoning of rosin barrel staves prior to coopering was found to require greater care in assembling the barrel. Coopering is more difficult, and many of the staves, particularly those having knots, break when the barrels are filled. Cooperative tests with the Forest Service on the value of the zinc-chloride treatment of rosin barrel staves for increasing the resistance of barrels to decay in open-air storage of rosin were completed. After 2 years of storage the barrels made of ¾-inch kiln-dried, zinc chloride-treated staves had broken down to the point where re-coopering was necessary.

During the year the naval stores station was visited by 762 persons, including 291 students and 13 instructors. Lectures and demonstrations were given to show the nature and purpose of the work. Eighteen demonstrations were given on the fire still, including 3 during training periods for technical turpentine workers, at which 75 workers were present. Eleven demonstrations were given of the method for cleaning gum and distilling it on the steam still. Information relating to naval stores production was given in 61 conferences and 167 letters.

Fifty-four turpentine operators in 4 States were visited by the accounting clerk, and 25 complete statements of operating costs were obtained. These data will be used in comparing the cost of each step and method of handling and processing the gum at the station with the cost of comparable operations in the industry.

Blueprints, line prints, and pencil sketches for still buildings, stills, and various kinds of equipment, to the number of 483, were distributed in response to requests from interested persons.

#### COOPERATION WITH STATES IN NAVAL STORES WORK

Cooperative agents continued to disseminate to naval stores producers in Florida and Georgia information concerning the improvements in practices and equipment for the production of naval stores developed at the naval stores station, by making personal contacts with producers individually or in groups.

Information on various subjects relating to the production of turpentine and rosin were given in Florida to naval stores operators, gum farmers, land-owners, and students in 343 personal contacts and 195 letters. The cooperative agent directed the erection of 18 turpentine fire stills and 5 still sheds according to plans and specifications approved by the Bureau, and he advised operators with regard to installation of 5 dehydrators, 15 covered separators, 7 recording thermometers, and 10 sets of rosin strainers. Twelve demonstrations of the improved method of stilling with regulatory equipment were given in the field on fire stills set in accordance with the Bureau's recommendations.

The cooperative agent made 180 visits to turpentine operators in Georgia, assisted in the setting of 25 stills, and furnished plans and specifications for and assisted in the construction of 13 model still plants. Several plants previously constructed with the aid of the cooperative agent have been copied by other naval stores operators; one has been copied for the fourth time. This shows the importance of these model still plants.

#### NAVAL STORES STATISTICS

Two statistical reports on naval stores were compiled and published during the year. One was issued on October 30, 1936, and the other on June 1, 1937. These reports give statistics on production, distribution, consumption, and stocks of turpentine and rosin, and they are designed to assist naval stores operators in adjusting production to probable demand. Tables of naval stores statistics for the seasons from 1900 to 1935 were also prepared for the Department's publication entitled, "Agricultural Statistics, 1937."

### OIL, FAT, AND WAX INVESTIGATIONS

#### LIQUID WAX FROM JOJOBA SEEDS

In last year's report it was stated that the seeds of *Simmondsia californica* had been found to contain over 51 percent of oil, which was exceptional, in that it contained 48.3 percent of unsaponifiable constituents. During the year, a comprehensive study was made of the material extracted from these seeds with petroleum ether. Although it has the appearance of a fatty oil, it is in reality a liquid wax. It is like the liquid wax obtained from the sperm whale, so-called sperm oil, in that it consists essentially of esters of unsaturated alcohols and unsaturated fatty acids. It differs from sperm oil, however, in the particular esters of which it is composed and also in its properties. When cooled to about 10° C. it solidifies. After heating for a short time at about 250°, the color changes from yellow to almost water-colored and remains so permanently. Its physical and chemical characteristics are as follows: Density at 25°, 0.8642; refractive index at 25°, 1.4648; iodine number (Hanus), 81.7; saponification value, 92.2; and acid value, 0.32. It contains about 1.64 percent of saturated fatty acids, 0.24 of palmitoleic acid, 30.3 of eicosenoic acid, 14.2 of dicosenoic acid, 14.6 of eicosenol, and 33.7 of dicosenol. Apparently it is entirely free from fatty oil or glycerides, since tests for glycerin were negative. Uses for this unique product remain to be discovered.

*Simmondsia californica*, commonly known as jojoba, or goat nut, is an ever-green shrub belonging to the box family, Buxaceae. It grows wild on hillsides in the somewhat arid sections of southwestern United States and western Mexico and is browsed by sheep and goats, especially during the dry winter season. Up to the present time this shrub is the only plant known to have seeds in which a liquid wax is elaborated in place of fatty oil.

#### OIL FROM NUTS OF JAPANESE "WOOD-OIL" TREE

Because of the growing importance of the seed oils of the genus *Aleurites*, the oil was expressed from the kernels of a 50-pound sample of *A. cordata* nuts, received from Japan through the Division of Plant Exploration and Introduction of the Bureau of Plant Industry, and its composition and properties were determined. The purpose of the Bureau is to obtain adequate comparative data on the composition and properties of the several industrially useful drying oils of related species through modern investigational methods. The Department is interested in the possibility of growing some of these species of nuts in the United States or its possessions.

The kernels, which constituted 63.8 percent of the weight of the nuts, contained 67.5 percent of pale-yellow oil which was somewhat similar in compo-

sition and properties to tung oil and only slightly inferior to that oil in drying properties. It contained 70.5 percent of elaeostearic acid, 18.5 of oleic acid, and 6.2 of saturated acids. Its physical and chemical characteristics were as follows: Refractive index at 25° C., 1.5060; saponification value, 190.2; acid value, 0.60; thiocyanogen value, 80.9; and iodine number (by calculation) 209.6. The properties of this oil make it valuable for use in the manufacture of paints, varnishes, and linoleum.

#### OIL FROM NUTS OF LUMBANG, OR CANDLENUT, TREE

A study was also made of the oil expressed from the kernels from about 100 pounds of lumbang nuts, or candlenuts, from the tree *Aleurites moluccana*, received from the Philippine Islands. Unlike the nuts of other *Aleurites* species, lumbang nuts have thick shells and the kernels adhere to them very tenaciously. The kernels, constituting about one-third of the weight of nuts, contained 69.2 percent of oil which differed from those of other *Aleurites* species in that it gave no test for elaeostearic acid. In composition and properties the oil is somewhat similar to linseed oil. It contained 26.2 percent of oleic acid, 39.6 of linoleic acid, 20.8 of linolenic acid, 4.4 of palmitic acid, 3.9 of stearic acid, about 0.1 of arachidic acid, and 0.3 percent of unsaponifiable matter. The chemical characteristics were iodine number (Hanus) 151.71, saponification value 190.8, and thiocyanogen value, 97.1. Commercial production of lumbang oil appears to be confined to the Philippines, where it is used chiefly in the manufacture of paint and varnish.

#### OIL FROM FRUIT SEEDS OF BRAZILIAN PALM

About 6 pounds of fruit from the Brazilian palm (*Syagrus coronata*) grown at Lake Worth, Fla., were submitted by the Bureau of Plant Industry for investigation. They were pale yellow and the size of small plums. They consisted of 47.5 percent of pulp and fiber and 52.5 percent of seeds or nuts, one to each fruit. The pulp contained a red oil which amounted to only 0.88 percent of the whole fruit. It is of no commercial interest because of the small yield.

For a study of the oil from the kernel, which has recently become of commercial interest, the oil was expressed from a 25-pound sample of kernels imported from Brazil. This contained 79.6 percent of saturated acids, 14.2 of unsaturated acids, and 0.3 percent of unsaponifiable matter. The following characteristics were determined: Iodine number, 14.7; saponification value, 256.9; thiocyanogen value, 12.78; Reichert-Meissl value, 5.93; and Polenske value, 18.38. There is some evidence that the oil contains an unusually large quantity of caproic acid which occurs only in traces and sometimes apparently not at all in coconut oil. It has the lowest solidification point (about 60° F.) observed in any palm-kernel oil. As there is no information in the literature on the composition of this oil, and numerous requests have been received for such information, further investigation is planned for the purpose of supplying it.

#### PHOSPHATIDES OF SOYBEAN OIL

In view of the rapidly growing importance of the domestic production and utilization of so-called vegetable, or soybean, lecithin preparations, it seemed desirable to make a further study of the soybean phosphatides. These phosphatides were extracted from soybeans by means of ethyl alcohol at 60° C. This treatment apparently removes phosphatides which are in combination with proteins and carbohydrates, as well as those present in a free state. The phosphatides were separated from extracted carbohydrates by solution in ethyl ether. Treatment of the concentrated ether solution with acetone yielded two acetone-insoluble and two acetone-soluble fractions. The acetone-insoluble alcohol-soluble fraction consisted of lecithin in combination with a betaglucoside. The acetone-insoluble alcohol-insoluble fraction consisted of cephalin in combination with another compound of about equal molecular weight. The third fraction, soluble in acetone, contained diamino-monophosphatides, and the fourth fraction, soluble in acetone, contained monoamino-diphosphatides.

### PROTEIN AND NUTRITION RESEARCH

#### PROTEINS OF WHEAT

The wheat kernel contains eight or more different proteins. Of these, only two, gliadin and glutenin, have been extensively studied. Little definite knowledge

is available regarding the others, either with reference to the relative proportions in which they are present in the kernel, or with reference to their amino acid content. The Bureau is frequently asked how much there is of particular amino acids in wheat, corn, oats, or other feed. The owner of a sheep ranch or fur farm who has read somewhere that cystine is important for the production of wool or fur may want to know how much cystine there is in certain foods for sheep or fur-bearing animals. Unfortunately, in most instances no satisfactory answer can be given. Usual methods for determining amino acids are applicable only to isolated, purified proteins. Interfering substances, such as carbohydrates and fats, preclude the application of these methods to the analysis of foodstuffs.

Realizing the importance of knowing how much of each nutritionally essential amino acid is present in various kinds of food or feed as used rather than in the purified proteins, the Protein and Nutrition Research Division developed a method for determining amino acids in staple foods, such as cereals, flours, meals, and seeds. During the last year this method was used to determine a number of nutritionally essential amino acids in whole wheat and its milling products, flour, middlings, and bran. Two papers were published giving information on the amino-acid content of wheat and on the effect of milling on the distribution of amino acids of wheat. Significant differences were found between the amino-acid contents of different varieties of wheat. Wheat varieties having a high nitrogen content (hard wheats) contain relatively higher percentages of the important amino acids than do those varieties having a low nitrogen content (soft wheats).

#### PROTEINS OF THE BLACK BEANS OF THE MAYAS

A study was made of the proteins of black beans obtained from Costa Rica through the Bureau of Plant Industry. It is reported that the diet of the Mayas, a sturdy and hardy tribe of Indians, consists almost entirely of corn and these beans. The proteins of this variety of bean and their amino-acid composition were studied to find out how well they supplement the proteins of corn in the diet and how they compare with the proteins of other varieties of beans previously studied. Two globulins were isolated, and their contents of the amino acids, arginine, histidine, lysine, cystine, tryptophane, and tyrosine, were determined.

The results of these studies showed that the proteins of these black beans share, in general, the same biological properties as the proteins of other botanically related legume seeds, namely, a pronounced deficiency of cystine and a high content of lysine; also, that they contain a liberal amount of tryptophane, another nutritionally essential amino acid. The proteins of corn, on the other hand, contain an adequate amount of cystine, but are deficient in lysine and tryptophane. When these black beans and corn are used together, there is a supplementary relationship between their proteins with respect to their amino acid content.

#### TRYPTIC DIGESTION OF CASEIN

In connection with the study, previously reported, on the rate of liberation of cystine from casein during laboratory digestion with trypsin, the question arose as to whether or not some of the cystine measured might have originated from the trypsin itself (containing 1.68 percent of cystine) as a result of autodigestion. In experiments carried out to throw light on this question it was found that when trypsin alone was digested a very significant amount of cystine was liberated. However, when casein which had been freed of cystine was digested with trypsin no cystine was found in the digest. This result indicates that in the presence of other protein in the digestion mixture the proteolytic activity of trypsin is exercised exclusively on the substrate without any appreciable autodigestion of trypsin taking place.

#### DECYSTINIZED CASEIN

Casein is generally used to supply the protein of basal rations employed in the biological assay of vitamins and as a control in experiments to evaluate the nutritional properties of proteins. It is also frequently used as a standard protein in certain determinations connected with protein and amino acid analyses. For these purposes the casein is first purified, frequently by dissolving it in dilute alkali and then reprecipitating with acid. The destructive effect of this treatment on the cystine has not been recognized generally or

taken into consideration in determining nutritional values. This fact undoubtedly has introduced errors into some past work and may account for a number of unexplained discrepancies which have been noted in publications in the field of nutrition and protein chemistry. Although alkali-treated casein is disadvantageous for some purposes, it occurred to the Bureau's protein chemists that casein which had been entirely freed of cystine by alkali treatment would be of value in the investigation of a number of problems.

By repeated treatment of casein with dilute alkali and reprecipitation with dilute acetic acid, a product was obtained which was substantially devoid of cystine. This product, as far as it has been analyzed, appears to have otherwise the same amino acid composition and the same distribution of nitrogen as the original untreated casein. A sufficient quantity of decystinized casein has been prepared recently, in order to allow a more thorough study of its chemical and physical properties and digestibility, and its uses in feeding experiments. The feeding experiments with this unique protein material are expected to elucidate a number of fundamental problems regarding the role played by the sulphur-containing amino acids, cystine and methionine, in nutrition.

#### SELENIUM IN TOXIC WHEAT

Work was continued on the properties and identity of the toxic organic selenium compound or compounds existing in wheat grown on seleniferous soils. Gluten separated from toxic wheat has been hydrolyzed with sulphuric acid and the resulting mixture of amino acids fractionated. One of the fractions, consisting chiefly of leucine and phenylalanine with smaller quantities of other amino acids present, contained most of the selenium which was originally present in the gluten hydrolyzed. This fraction was prepared repeatedly, and various methods were used in trying to separate the selenium compound from the amino acids.

In one method the selenium-containing fraction dissolved in water was subjected to direct electric current under high voltage. Under certain conditions the selenium, together with some of the leucine and tyrosine, migrate to the negative electrode. Although a clear-cut separation of selenium from amino acid was not accomplished, a mixture containing a higher concentration of selenium was obtained. Conditions governing migration of the selenium compound, such as temperature, pH value of the solution, voltage, and current, were studied.

In another method, a solution of the selenium-amino acid fraction was treated with mercuric chloride, whereby a precipitate was formed carrying practically all of the selenium. This product, containing about 2 percent of selenium, is only very slightly soluble, even in comparatively strong sulphuric acid or hydrochloric acid solutions. Because of this unusual property a successful decomposition of the salt and separation of the selenium compound from the mercury has not yet been accomplished. Qualitative tests for sulphur in the selenium-mercury precipitate gave negative results. This lends support to the belief that selenium in the wheat protein is not merely a substitute for sulphur in the amino acids cystine or methionine, as frequently has been suggested.

#### EFFECT OF STORAGE ON PROTEINS OF SOYBEAN OIL MEAL

It has been observed that the proteins of some seed meals may lose about one-half of their nutritive value within a few weeks after grinding. These observations have suggested that similar changes may occur when unground grains or other seeds are held in storage.

An investigation has been started, therefore, in which chemical and biological experiments are made to determine the nature and extent of changes which take place in the proteins of grains and other seeds and their mill products, when stored for periods ranging from 1 month to 2 years or longer. Particular attention is given to storage conditions, such as temperature, moisture, exposure to light, type of container, and fat content, which may hasten or retard deterioration.

Chemical analyses and tests on freshly ground soybeans and on the same meal after storage periods of 1 month and 3 months showed that within 1 month changes took place in the chemical properties, solubility, and digestibility of the proteins, which indicated an alteration in the integrity of the protein structure. These changes occurred to a greater extent in meal stored at ordinary temperature than in that stored at refrigeration temperature. After

3 months of storage, the changes were more pronounced. Sufficient data have not yet been obtained to justify the drawing of conclusions regarding the nature and ultimate extent of the changes caused by storage.

#### VITAMIN B COMPLEX IN SOYBEANS

In order to develop information required in formulating the rations to be used in feeding experiments, a study was made of the vitamin B complex of Mammoth Yellow soybeans harvested in the late fall of 1936. If the soybeans contained an adequate amount of the vitamin B complex it would be unnecessary to supplement the soybean rations with vitamins from other sources. It was found that sufficient vitamin was supplied by this particular variety and lot of soybeans when the soybean oil meal constituted 20 percent of a ration otherwise free from vitamin B complex. No improvement in rate of growth was observed when the soybean oil meal content of the rations was increased beyond 20 percent.

#### VITAMIN STANDARDS

The Bureau has continued to distribute the international vitamin standards received from the Health Organization of the League of Nations. These standard samples have been issued on request to vitamin investigators in the United States, working in colleges, universities, and agricultural experiment stations.

#### VITAMIN ASSAY OF ROYAL JELLY

Royal jelly is the special food which bees feed the larvae, in order to develop queens for their colonies. It has the property of greatly stimulating growth in bees and in developing the female sex characteristics, but the component to which these remarkable properties may be attributed is not known. Some work has been done outside the Department on the vitamin E content of royal jelly, but the results obtained are conflicting. Studies on the vitamin assay of royal jelly have recently been started at the request of the Bureau of Entomology and Plant Quarantine. The results are expected to be useful in connection with the development of synthetic rations for use in studying problems related to bee culture.

#### ALLERGENS OF AGRICULTURAL PRODUCTS

A study of allergens of agricultural products was carried on during the year as a part of the Department's program of basic research in agriculture, provided for in the Bankhead-Jones Act of June 29, 1935.

Clinical facilities were organized at Providence Hospital in Washington, for determining the potency and specificity of fractions prepared in the laboratory from allergenic products. Approximately 300 patients of the 1,200 who have visited the allergen clinic have been registered and classified for testing with materials of special significance. About 70 patients have been under continual study, in order to determine the activity of fractions derived from milk, ragweed, buckwheat, cotton linters, and cottonseed.

Experiments were made to determine if milk may contain allergens derived from the dairy ration. Ragweed allergen was not detected in the milk of cows fed on ragweed tops or in fractions prepared from this milk.

New cotton linters were found to be free from soluble allergens affecting individuals who are sensitive either to house dust or to cottonseed. Storage experiments with small quantities of linters under various controlled conditions are in progress, to determine if this material generates a specific dust allergen on aging. Extracts prepared from cotton linters taken from a bale which had been in storage for 9 years were consistently negative when tested on clinical patients sensitive to dust and cottonseed allergens. These extracts contained only a trace of nitrogen.

Fractionation of allergen extracts from cottonseed, so as to concentrate the active principle while decreasing the total protein content, was accomplished by subjecting the solutions to the action of a direct current of electricity which caused the cottonseed allergen to migrate toward the positive electrode. An active fraction was obtained which contained only a small proportion of the nitrogen in the original crude extract. When the current was allowed to act for a long time all fractions were completely inactivated. When an extract of ragweed pollen was similarly subjected to the action of an electric current, the allergen migrated toward the negative electrode. The ragweed allergen was more stable than the cottonseed allergen when the current was allowed to act for long periods.



Preparations containing allergens of buckwheat, wheat, and cottonseed were used as substrates for comparing the enzymic activity of blood from normal and allergic subjects. Measurable differences were observed for the total enzymic activity of blood from different subjects. Whether these differences are related to the constitution of allergens cannot be concluded from the data now in hand.

## CHEMICAL ENGINEERING RESEARCH

The work of the Chemical Engineering Research Division during the year has included investigations on the causes and prevention of fires and explosions in the handling, storage, and processing of agricultural materials and of other accidents of a chemical nature on a farm and in a rural community; also engineering design, drafting, and development services to other divisions of the Bureau.

### AGRICULTURAL FIRES

One of the main causes of fires on farms is the spontaneous ignition of hay and other organic materials. The Bureau has found that one of the principal factors contributing to spontaneous heating and possible ignition of hay is a high moisture content, and that purely chemical, as well as microbial, action is involved. Even a moderate degree of self-heating in hay results in the loss of organic substance which reduces its feeding value.

Laboratory and field investigations were continued on the causes, results, and prevention of spontaneous heating in hay. A large-scale experiment on first-cutting alfalfa hay (1936 crop) was completed in the experimental barn at the National Agricultural Research Center, Beltsville, Md. The primary object of this experiment was to determine the effect of an elevated openwork floor and open alleyway on the rate of drying of undercured long hay in storage and dissipation of the heat produced. The mow was divided into two compartments, each 12 by 23 feet, separated by a wooden partition of 2 by 4's covered on each side with insulating board seven-eighths of an inch thick. One compartment had an elevated false floor made of 2 by 4's set on edge with 4 inches between faces across 2 by 8's set on edge with about 3 feet between centers and notched so that the upper edges were flush with those of the 2 by 4's. There was an open alleyway at one end of each compartment. About 12 tons (eight loads) of hay were put in each compartment as uniformly as possible, and special care was taken to have the hay in the two compartments as similar as possible. Each load was sampled for moisture content. The average moisture content of the hay in the ventilated compartment was 36.82 percent; that of the hay in the other compartment was 38.62 percent. The height of hay in the ventilated compartment was 15 feet; that of the hay in the other compartment was 14½ feet. The hay was left in the mow for practically 8 weeks in June and July. During this period the temperatures of the hay in the two compartments, in corresponding positions at different levels, were recorded, and the relative upward velocities of air near the surface of the hay in the two compartments were observed, by means of suitable instruments. The results of this experiment indicated that the use of an openwork support in the storage of long hay in mows of moderate size is desirable as a means of reducing deterioration due to heating. The advantage of using such a false floor in mows of great height is doubtful, because dense packing of the haymows might prevent adequate aeration.

A member of the Chemical Engineering Research Division's staff conferred with representatives of two State agricultural colleges and one State agricultural experiment station with regard to current and contemplated investigations on spontaneous heating and ignition of hay, gave information and advice to two manufacturing firms which are experimenting with special structures and containers for chopped hay, and twice addressed the American Society of Agricultural Engineers on the subject of spontaneous heating and ignition of hay.

At the request of farmers in the Ohio River Valley in the vicinity of Point Pleasant, W. Va., members of the chemical engineering staff investigated spontaneous heating in haymows, which resulted from wetting by the floodwaters of January 1937. Temperatures as high as 65° C. (149° F.) were found in several large mows. The hay was removed and scattered, in order to prevent spontaneous heating to the point of ignition. In several instances, soybean hay in bales had heated so severely that it had to be dumped into the water to prevent an outbreak of fire.

Detailed instructions for handling mows and stacks of heating hay were made available.

Experiments were continued in the laboratory to determine whether the easily oxidizable unsaturated products previously indicated as present in heating hay resulted from heat alone. It was found that heating of hay in an inert atmosphere results in an increase in the oxygen absorption by the hay. Progress has been made in the study of the fatty substances of hay, which largely are lost as the result of self-heating. Experiments to determine the gaseous decomposition products of hay heated to various temperatures in an atmosphere of nitrogen, with special reference to the formation of carbon monoxide, are still in progress.

Members of the Bureau's chemical engineering staff cooperated with organizations interested in fire prevention, by participating in conferences and meetings, and in the work of committees on which they held membership. These organizations included Underwriters' Laboratories, Chemical Fire Extinguisher Association, State Association of Mutual Insurance Companies of Michigan, Mutual Insurance Companies' Union of Indiana, American Society of Agricultural Engineers (committee on fire prevention and protection), National Fire Waste Council of the Chamber of Commerce of the United States, and its agricultural committee, and the National Fire Protection Association (committees on farm fire protection and spontaneous heating and ignition). The National Fire Protection Association was assisted in the preparation of its spontaneous heating and ignition committee's report on Spontaneous Heating and Ignition of Coal and Other Mining Products, in the revision of its farm fire protection committee's report on Rural Fire Departments, Equipment, and Organization, and in the preparation of the same committee's tentative report covering desirable provisions in State laws on rural fire protection, suggested model laws establishing fire-protection districts and providing fire protection and proposed model ordinance for the organization and administration of volunteer fire departments.

#### DUST EXPLOSIONS

Progress has been made in reducing the dust-explosion hazard in industrial plants handling or processing agricultural products. As a result of the research work carried on in the laboratory and at the dust-explosion testing station at Arlington Experiment Farm, many precautionary measures have been developed and made available in the form of recommended safety codes for the prevention of dust explosions.

Thirteen explosions in industrial plants, involving dusts from agricultural materials were investigated by the Bureau during the past year. Four of these were caused by grain dust, two by cotton floc or lint, six by wood dust, and one by dust from powdered drugs. These explosions resulted in the death of 14 men, injury to 43, and property damage amounting to about \$1,147,000. The most serious explosion occurred in a corn milling plant at Milwaukee on April 10, 1937. It resulted in the death of 9 men, injury to 24, and a property loss of about a million dollars. In five of the explosions the property losses ranged from \$10,000 to \$40,000, and in the others they ranged from \$1,000 to \$5,000.

The recent establishment of new industries using agricultural products and the increased utilization of byproducts have broadened the field of research and experimental work necessary to develop the additional safety measures required for the protection of these industries from dust explosions. The rapid growth in the production of soybeans has encouraged the erection of plants for their industrial utilization. As a result, there has been an increase in the number of establishments subject to the hazard of possible ignition of dust or volatile solvents during the milling and processing of the beans. Two explosions in soybean processing plants in 1935 resulted in the loss of 13 lives, injury to 47 persons, and a property loss of over \$600,000. These explosions indicated the importance of developing and applying safety measures in plants of this kind, and the Chemical Engineering Research Division is working on this problem.

Tests to determine the breaking strength of glass used as explosion vents were continued during the year. Special attention was given to the possibility of scoring glass in order to reduce its resistance to pressure from within a building without seriously lowering its resistance to wind pressure from without. Scoring with a diamond proved more effective than with a steel glass cutter. Tests are under way to determine the effect of prolonged exposure to weather on the breaking strength of scored glass.

During the year experiments were continued to determine the conditions under which dust clouds may be ignited by metallic sparks.

A number of dust samples submitted by manufacturing firms and representatives of organizations interested in prevention of accidents were tested during the year to determine their explosibility. Those which were found to form explosive mixtures with air included soap powders, pulverized bagasse, grain-elevator dusts, carbohydrate adhesives, casein dust, wood dusts, soybean flour, soybean-protein dust, wood-pulp fines, powdered vegetable char, and spray-dried sulphite cellulose waste. Samples of powdered stearic acid when mixed with air were very explosive.

The results of the Bureau's work on prevention of dust explosions were presented at 11 firemen's training schools (in 10 States and the District of Columbia) and before 2 firemen's conventions; the fire departments of 8 western cities; 11 student groups at high schools, colleges, and universities; 2 civic organizations; and the annual gathering of the 4-H Clubs in Washington, D. C. In addition, addresses were given before the Factory Inspector's School at Chicago, the Chicago Engineers' Club, the engineering section of Chicago Safety Council, the American Soybean Association, and a conference of fieldmen of a southern grain company.

In connection with their work on dust-explosion prevention, members of the chemical engineering staff cooperated with national organizations by active participation in the work of committees on which they held memberships or before which they were requested to appear. The dust-explosions hazards committee of the National Fire Protection Association, of which the chief of the Chemical Engineering Research Division is chairman, was assisted in amending the safety code for the prevention of dust explosions in grain elevators to provide for the use of dust-proof enclosures for conveyor belts and in the formulation of a general safety code embodying the fundamental principles for the prevention of dust explosions in industrial plants. This new code is intended for the many plants which have some dust-explosion hazard but for which no specific code has been prepared. The blower and exhaust systems committee of the National Fire Protection Association was assisted in revising the code covering the installation of blower and exhaust systems for dust, stock, and vapor removal and in drawing up additional regulations for the installation of ventilating and air-conditioning systems. The static electricity committee of the same organization was assisted in the preparation of a safety code designed to call attention to the hazard of static electricity in industry and containing recommended safe-practice regulations. The conveyor committee of the American Standards Association was assisted in the preparation of a safe-practice code for installation and use of conveyors.

#### SILO GAS ACCIDENT

Members of the chemical engineering staff investigated the accidental death on September 3, 1936, of a mother and two daughters in a pit silo on a farm near Poplar Springs, Md. The younger child collapsed while playing in the silo containing fresh silage, and the mother and other child lost their lives in attempting to rescue the little girl. The investigation indicated that these deaths were caused by suffocation in an atmosphere deficient in oxygen. Samples of the gas in the silo were collected 16 hours after the tragedy occurred, during which period another load of silage had been added. The gas taken at the surface of the silage contained about 10.5 percent of carbon dioxide and between 18 and 18.5 percent of oxygen. A sample of gas taken 2 feet below the surface of the silage contained over 91 percent of carbon dioxide and not more than 1.5 percent of oxygen.

This case emphasizes the dangerous condition which may exist in silos, especially of the pit type, during the storage of fresh silage, when large quantities of carbon dioxide gas are evolved as a result of the fermentation process.

A warning with recommended procedure for preventing such accidents was issued through the Department's press service and radio service.

#### GAS EXPLOSION IN TEXAS SCHOOL

At the request of Gov. James V. Allred of Texas, an investigation was made of the consolidated-school-building explosion at New London, which occurred on March 18, 1937. Definite information was obtained as to the source of ignition for the gas which had accumulated under the main part of the building. The report of this investigation, which was transmitted by the Department to Senator Connally of Texas on April 14, was printed as a Senate document (75th Cong., 1st sess., Doc. No. 56).

### SERVICE WORK

One member of the chemical engineering staff was detailed during the entire year to the Naval Stores Research Division for engineering work in connection with technological investigations on the production and utilization of turpentine and rosin.

In collaboration with the agricultural byproducts laboratory of the Industrial Farm Products Research Division, designs were prepared for the special metal equipment to be used in pilot-plant experiments on the production of paper pulp from farm wastes and on the production of organic acids from sugars by mold fermentation. Plans and specifications were prepared for laboratory equipment for rooms in the South Building, and some time was given to the preparation and revision of estimates for construction and equipment of proposed buildings at Beltsville.

### FERTILIZER RESEARCH

In the Bureau's fertilizer investigations, fundamental research is conducted on the ultimate structures and the physical and chemical properties of the elements and compounds contained in fertilizer materials, on the kinetics of the chemical reactions involved in the fixation of atmospheric nitrogen and other processes of fertilizer manufacture, and on the mechanism of nitrogen fixation by living organisms. Laboratory procedures are devised for the preparation of potential fertilizer materials in quantities sufficient for studies of their reactions with others and for vegetative tests, in cooperation with other units of the Department and with State agricultural experiment stations. New processes for the manufacture of fertilizer are tested on a semicommercial scale which serves to demonstrate their feasibility to the industry. Economic and statistical studies on the production and utilization of fertilizer are made also.

#### CATALYSTS IN NITROGEN AND PHOSPHATE FERTILIZER PRODUCTION

Studies on the kinetics of the synthesis and decomposition of ammonia were continued for the purpose of ascertaining how iron synthetic-ammonia catalysts function and the mechanism through which promoters modify the properties of such catalysts. Variations in the particle size of the catalyst were found to have no influence on its activity. This indicates that not merely the geometric surface, but the internal, or inner, surface of the particles is of importance in catalysis. Endeavors to modify the surface characteristics of an iron synthetic-ammonia catalyst, singly promoted by a 10.2-percent content of alumina, by reduction and deposition of tiny active iron crystals thereon from iron pentacarbonyl were unsuccessful because of inability to cause decomposition of carbonyl on the inner surface of the catalyst with the apparatus employed. Experiments which aimed at the incorporation of potash as a second promoter into the surface of an iron catalyst that already contained alumina met with success; the sorptive capacity of the resultant catalyst indicated that about 50 percent of the surface was covered with an alkaline promoter, and the energy of activation for ammonia decomposition was found to have increased from 25,000 calories per molecule of ammonia, which is apparently characteristic for the iron-alumina catalyst, to about 40,000 calories per molecule of ammonia. The latter value is about the same as that found for doubly promoted catalysts prepared according to the usual procedure by incorporating both alumina and potassium oxide in molten magnetic iron oxide and reducing the product.

The method for measuring the surface areas of iron synthetic-ammonia catalysts, by determining the adsorption isotherms of gases near their boiling points, described in the report of 1935 and mentioned in the report of 1936 as having been found applicable to the determination of the surface areas of other finely divided materials, such as pumice, glaucosil, chromium oxide gel, and crystalline chromium sesquioxide catalysts, apparently can be applied also to the measurement of the relative and absolute surface areas of soils and soil colloids. By use of this procedure, surface-area values for samples of Barnes soil, Barnes soil colloid, Cecil soil, and Cecil soil colloid were found to be equivalent to 4.2, 3.2, 8.0, and 5.5 acres per pound, respectively. Calculated values for the colloid content of soils, obtained by use of such surface-area values, were in good agreement with the values obtained by mechanical analysis. By means of the same method, alumina, when present as a promoter in an iron synthetic-ammonia catalyst, has been shown to accumulate on the

surface of the catalyst in such a manner that a small percentage thereof in the catalyst covers a relatively large fraction of the surface; thus, 10.2 and 1.03 percent of alumina in two samples covered 60 percent and 35 percent of their respective surfaces.

Continuation of the investigations on the oxidation of phosphorus vapor with carbon dioxide showed the lower oxide of phosphorus, mentioned in last year's report, to be phosphorus tetroxide. Efforts to convert this to phosphorus pentoxide by oxidation at temperatures below those at which the carbon monoxide, the product desired for the manufacture of hydrogen for ammonia synthesis, also would burn were without success. It was found, however, that the passage of the original mixture of phosphorus vapor and carbon dioxide through a layer of phosphate rock heated to 1,000° C. resulted in complete oxidation of the phosphorus to the pentoxide which combined with the rock to produce calcium metaphosphate, a compound containing in excess of 65 percent of  $P_2O_5$ . These investigations have, therefore, led to a process whereby phosphorus-carbon monoxide mixtures, such as are obtained in electric furnaces for the reduction of phosphate rock, can be made to react with carbon dioxide in the presence of additional phosphate rock to produce carbon monoxide and calcium metaphosphate, which when finely ground appears suitable for use as a concentrated phosphatic fertilizer; the process conserves all the carbon monoxide present in the furnace gases and produces an equal additional quantity.

#### PHYSICAL CONSTANTS OF GASES AND FERTILIZER SALTS

In continuation of the program to obtain by direct experimental determination a knowledge of the compressibilities of ammonia, hydrogen, nitrogen, and their mixtures for the calculation of volumes, pressures, viscosities, thermal conductivities, specific heats, and other thermal quantities under the variety of conditions encountered in the synthesis of ammonia, the compressibilities of an 87:13 hydrogen-nitrogen mixture at 0°, 25°, and 50° C. and at pressures from 25 to 1,000 atmospheres and of a 50:50 hydrogen-nitrogen mixture at 0°, 25°, 50°, 100°, 200°, and 300° over the same pressure range have been measured. A knowledge of the ammonia that is left in the vapor phase at different temperatures and pressures when the ammonia is condensed out from gaseous mixtures is important; consequently, previous measurements were extended to include determinations of the vapor content of ammonia in ammonia-hydrogen mixtures at 0° and 100° at various pressures. The solubilities of hydrogen in liquid ammonia at 0° and of nitrogen in liquid ammonia at 0° and 50° were measured at pressures ranging from 50 to 1,000 atmospheres.

Vapor pressure determinations were made for the two crystalline modifications of phosphorus pentoxide and for the liquid. The partial pressure of water vapor as a result of the dissociation of hydroxyapatite was found to be less than 1 atmosphere at 1,500° C. The structures of phosphorus trioxide and phosphorus pentoxide were determined by electron diffraction. The citrate-soluble materials formed when phosphate rock is calcined were found to be alphanitric calcium phosphate, silico-carnotite, and a compound having the approximate composition,  $3Ca_3(PO_4)_2 \cdot 2Ca_2SiO_4$ . When these materials are annealed in an atmosphere of steam below 1,200°, reversion to citrate-insoluble forms that have the structure of apatite takes place in many instances, but they are resolubilized with formation of the initial phases on reheating at 1,400° in an atmosphere of steam.

Work in cooperation with the Bureau of Entomology and Plant Quarantine showed that all calcium arsenate samples known to injure the foliage of apple trees contained dicalcium arsenate dihydrate. In cooperative work with the Bureau of Plant Industry to determine the effects of X-radiation on the history of corn smut, dosages of 100,000 R units were found to produce a distortion of the promycelium and to reduce greatly the number of sporidia, although these were normal in appearance. Extension of the investigations conducted with the latter Bureau on the effect of X-rays on corn seeds and tobacco plants, mentioned last year, disclosed that the delayed killing observed for corn occurred also with tomatoes, wheat, buckwheat, and pigweed, and that the germination of rice was stopped by a dosage greater than 35,000 R units.

Infrared absorption spectra characteristic of nitrous acid and nitric acid molecules have been found, that allow a quantitative study of the nitrous acid equilibrium and give promise of contributing directly to the elucidation of the structure of the nitrous acid molecule. In cooperation with the Bureau

of Standards, the infrared absorption spectra of a series of hydrocarbons having high molecular weights were determined.

The mass-spectrographic investigation of the abundance ratio of potassium isotopes in nature showed that bone marrow is high in the heavy isotope and that heart muscles are low in the radioactive isotopes.

#### NITROGENOUS FERTILIZER MATERIALS

In continuation of previously reported work for the purpose of combining inorganic nitrogen with waste organic materials to obtain products containing organic nitrogen, it was found that, in varying the ratios of ammonia to peat over a range from 0.1 to 2.0, the maximum amount of soluble nitrogen and the highest activity for the insoluble nitrogen were obtained with a ratio of 0.3 or 0.4. Peat samples ammoniated at 130° C. under 500 to 600 pounds pressure, with 40 percent of water and a ratio of ammonia to peat of 0.3 or 0.4, gave products which, in a series of pot tests, gave yields of carrots equaling those obtained with urea and cottonseed meal. The total nitrogen in the products was about 6 percent, and chemical tests showed relatively high activity for the insoluble nitrogen, of which 33 to 40 percent nitrified in the soil in 8 weeks.

In connection with the studies on ammoniation, it was found that treatment of dicyanodiamide with liquid ammonia at 155° C. resulted in a 75- to 80-percent conversion to melamine, a compound that contains about 68 percent of nitrogen. The nitrate, sulphate, and phosphate salts of this compound were prepared. Preliminary water-culture tests for their plant-food value gave results that were encouraging, but later similar tests and pot tests threw considerable doubt on the value of the melamine nitrogen as a plant food.

Preliminary studies on the hygroscopicities of the compounds of urea with magnesium sulphate and with magnesium nitrate, mentioned in last year's report, indicate that the compound with magnesium nitrate is very hygroscopic, and the compounds with magnesium sulphate much less so. The addition of 2 to 4 percent of driers such as calcium carbonate or oxide and magnesium oxide, improved the properties so that the sulphate compounds withstood humidities of 75 to 80 percent without caking. In similar experimental work on crystalline urea, no drier was found to be entirely satisfactory for reducing its caking tendency. An addition of 2 to 4 percent of lime proved most effective for this purpose.

#### BIOCHEMICAL AND ORGANIC NITROGEN INVESTIGATIONS

In studies of nitrogen fixation with additional strains of *Azotobacter*, conducted similarly to the studies with *A. vinelandii*, previously reported, a particularly interesting strain was found that was very low in nitrogen content and formed ammonia very slowly on the elimination of carbohydrate. The optimum temperature for its growth was higher, and its rate of growth was faster than in the other strains tested, and it preferred free rather than fixed nitrogen. In further experimental work, dealing with the effect of molybdenum on *Azotobacter*, nitrogen fixation was shown to be increased by this catalyst from 100 to 500 percent, depending on the strain of *Azotobacter* used. The molybdenum had no appreciable effect on the utilization of several fixed nitrogen compounds tested, which indicates that it serves as a catalyst in the fixation process itself, entirely apart from growth.

An extensive series of experiments was completed, in which an attempt was made to determine the conditions under which the root nodules of legumes may excrete nitrogen into the sand or soil, in which they are growing. With possibly one exception, no such nitrogen excretion was obtained. The results, therefore, like those of the preliminary studies mentioned last year, failed to confirm the findings of a Finnish worker who has repeatedly reported large excretions of aspartic acid and another unidentified nitrogen compound. Although these experiments do not disprove that the root nodules of legumes excrete nitrogen under some conditions, they show that heavy excretion of nitrogenous compounds does not always occur under good growing conditions.

A number of pure culture studies with the blue-green alga, *Nostoc muscorum*, previously isolated from the soil, showed that this organism fixes nitrogen from tenfold to twentyfold more rapidly than the few nitrogen-fixing blue-green algae previously studied by various workers, as high as 10 mg of nitrogen being fixed in 45 days per 100 cc of culture medium containing no carbohydrate. Nitrogen fixation also takes place slowly in the dark if a suitable source of

energy, such as glucose, is supplied. Details of growth conditions, such as composition of the medium, need for calcium and magnesium and various trace elements, and optimum light intensity, were determined. The studies indicate this organism to be of considerable economic importance in soils, since it adds both nitrogen and organic matter.

The composition of *Azotobacter vinelandii* was found to vary markedly when it was grown under different conditions; for example, if the only form of nutrient is atmospheric nitrogen the anhydrous bacteria contained 7.1 percent of nitrogen, 12.7 of lipid matter, and 4 of wax, as compared with 10.7 percent of nitrogen, 7.3 of lipid matter, and 10 of wax when grown on urea. Azotobacter nucleic acid prepared from organisms grown on atmospheric nitrogen was hydrolyzed under various conditions to isolate and identify its components; the bases present were cystosine, uracil, adenine, and guanine, and the carbohydrate proved to be ribose.

In a continuation of work on fixing nitrogen by reaction with organic substances apart from life processes, certain reactive unsaturated groupings were found to be reduced by 9-hydroxy- and 9-amino-fluorene derivatives in the presence of such catalysts as alkali or organic bases. Thus, azobenzene is converted to hydrazobenzene and dibiphenylene ethylene to the ethane derivative, whereas diazofluorene is reduced in two different ways, fluorene and nitrogen being formed in one reaction and fluorenone hydrazone in the other.

### PHOSPHATES

Since the chemistry of defluorinated phosphates, such as calcined phosphate and fused phosphate rock, involves compounds about which little or no information is available, a study was initiated of the system  $\text{Ca}_3\text{P}_2\text{O}_8\text{-CaO-SiO}_2\text{-H}_2\text{O}$  at temperatures up to  $1,500^\circ\text{C}$ . When synthetic mixtures that fall within the range of composition of commercial phosphate rock were heated at  $1,400^\circ$ , the citrate-soluble compounds formed corresponded to those found in calcined phosphates obtained by heating phosphate rock in the presence of steam to  $1,400^\circ$ . At  $1,400^\circ$  the tricalcium phosphate forms an extensive series of solid solutions with calcium orthosilicate, and the silicocarnotite also forms solid solutions with the tricalcium phosphate.

A study of the boron content of natural phosphates showed that this element occurs in small quantities in phosphate rock from deposits throughout the world. In 40 samples of domestic phosphate rock, the content of acid-soluble boron trioxide ranged from less than 10 to 144 parts per million. In general, phosphate rock also contains acid-insoluble boron.

Investigations were continued on the factors affecting the reversion of the citrate-soluble phosphorus of calcined phosphate to the citrate-insoluble condition at temperatures below  $1,400^\circ\text{C}$ . The degree of reversion varies with the nature of the furnace atmosphere, the temperature at which the calcined phosphate is annealed, and the chemical composition of the calcined phosphate. It was found that calcined phosphates can be divided into three general classes, namely, (1) those that undergo no marked reversion in either a moist or a dry atmosphere, (2) those that show marked reversion in a wet atmosphere only, and (3) those that suffer marked reversion in both wet and dry atmospheres. When reversion takes place in both wet and dry atmospheres, it is usually much greater in the former. Reversion may occur throughout the temperature range of  $200^\circ$  to  $1,300^\circ$ , but it usually is greatest from  $700^\circ$  to  $1,250^\circ$ . The reverted phosphorus can be transformed into the citrate-soluble condition by reheating the material for a few minutes to  $1,400^\circ$  and cooling the product quickly. Although reversion of the phosphorus occurs very rapidly under laboratory conditions, examination of a number of samples prepared on a semicommercial scale indicated that reversion will not be a serious factor in the manufacture of the material. Reversion to the citrate-insoluble form is accompanied by the formation of phosphates that have an apatitelike structure.

Further studies on the plant-food value of calcined phosphate, in cooperation with a number of State experiment stations, indicated, in general, that the material, when ground to pass a 40-mesh sieve, is as effective per unit of  $\text{P}_2\text{O}_5$  as superphosphate in promoting the growth of plants in neutral and acid soils, but it appears to be slightly less effective when applied to highly alkaline, calcareous soils. The phosphorus in reverted calcined phosphate is somewhat less available to plants than is the phosphorus in the rapidly cooled, unreverted product.

Pot tests with fused phosphate rock, which is similar to calcined phosphate in its chemical and physical properties, and calcium metaphosphate indicated that,

in general, these materials are good sources of phosphorus for plant growth. The importance of removing water-soluble compounds prior to the determination of citrate-insoluble phosphorus in fertilizers was demonstrated.

Preliminary experiments on the fixation of organic and inorganic phosphates from water solutions by soils indicate that the amount of organic phosphate removed from the solution varies with the soil used, the pH of the solution, and the time of contact. A soil high in colloidal matter, such as Cecil clay, removes the major portion of the inorganic as well as organic phosphate whereas a soil low in colloids, such as Norfolk loam, removes very little of either when the time of contact is only a few days. The quantity of organic phosphate removed from a water solution by Norfolk loam increases considerably as the time of contact is lengthened. In determinations by standard fertilizer methods of the amounts of organic phosphate fixed by soil, several widely different kinds of water-soluble organic phosphates were found to be entirely fixed by the soil used in the experiments.

#### POTASH SALTS AND BYPRODUCTS

Work has been continued on the utilization of dilute nitrosyl chloride gas, a byproduct obtained in the preparation of potassium nitrate by direct treatment of solid potassium chloride with gaseous nitrogen oxides produced by the oxidation of ammonia with air. Experiments on the absorption of this gas by moist phosphate rock for the production of available phosphates have shown that chlorine and nitrogen are absorbed by rock-water mixtures containing up to 50 percent of water in approximately the ratio of 2 mols of chlorine to one of nitrogen for nitrosyl chloride concentrations of about 4 percent. Because of the difficulty in maintaining fresh absorption surfaces with these mixtures, absorption ceased before complete decomposition of the rock was obtained. With larger proportions of water present, practically complete decomposition of the rock was obtained, but there was almost no preferential absorption of chlorine. In all instances the products obtained required drying. In some instances the removal of comparatively large amounts of water was necessary to produce a fertilizer material of satisfactory physical properties.

In further work on the preparation of potassium metaphosphate, a prospective fertilizer material that analyzes 40 percent of  $K_2O$  and 60 percent of  $P_2O_5$ , equimolar mixtures of potassium chloride and phosphoric acid were heated at various temperatures up to  $900^\circ C$ . The formation of the metaphosphate was favored by the higher temperatures, and the water-soluble portions of the product were found to contain unreacted chloride, together with phosphate in ortho, meta, and pyro forms. A product prepared from a commercial muriate that contained small amounts of sodium and sulphate impurities was found to contain 41 percent of its potash in water-soluble form. Products prepared similarly from reagent quality materials were practically insoluble. Aqueous solutions of sodium metaphosphate were found to exert a solvent effect on potassium metaphosphate and, in some instances, to result in thick viscous solutions which were remarkable because they contained less than 3 percent of total solids. Solutions of normal viscosity, however, were obtained by dissolving a fused mixture of sodium and potassium metaphosphates. The existence of potassium metaphosphate in one glassy and two crystalline forms was confirmed. The glassy form is water soluble and is the most difficult to prepare. It was obtained only by exceedingly rapid cooling of molten metaphosphate. With less rapid cooling, the metaphosphate crystallized in an insoluble form. The other crystalline form is water soluble and was obtained by neutralization and low-temperature evaporation of a solution of metaphosphoric acid.

In efforts to utilize the hydrochloric acid obtained as a byproduct when potassium chloride is converted into other potash salts, it was discovered about 2 years ago that this acid reacts with phosphate rock to form monocalcium chlorophosphate. Continued studies on this new compound showed that it is the product of a second-order reaction between calcium chloride and phosphoric acid, in which one-half of the chlorine in the calcium chloride is evolved as hydrochloric acid. Macroscopic crystals of monocalcium chlorophosphate sufficiently large for petrographic determination were obtained by evaporating and digesting at the boiling point aqueous, alcoholic, and glacial acetic acid solutions of calcium chloride and phosphoric acid. Similar procedures with barium chloride-phosphoric acid and magnesium chloride-phosphoric acid solutions failed to yield the analogous barium and magnesium salts. Monocalcium chlorophosphate undergoes both hydrolysis and solution in contact with water and alcohol. When treated with aqua ammonia or ammonia gas it is readily



ammoniated, and, because its citrate solubility is decreased only slightly, it is presumed that it forms ammonium chloride and dicalcium phosphate. On heating to 500° C. it decomposes to calcium pyrophosphate, losing both hydrochloric acid and water.

Vegetative tests on potassium metaphosphate and monocalcium chlorophosphate have shown both to be promising new fertilizer materials.

#### MIXED-FERTILIZER TECHNOLOGY

In continuation of the investigations on the granulation of fertilizer mixtures and materials, studies were made of the effects of temperature and moisture content on the granulation of 260 mixtures and individual materials and also of the effect of variations in particle size of the initial materials on the efficiency of the granulating process. Mixtures that consisted largely of inorganic materials were found to granulate more readily and with a lower moisture content than mixtures that were comparatively high in organic materials. Determinations of particle size previous to the granulation of mixtures and materials showed that the efficiency of granulation was reduced from 89 to 74 percent when the proportion of initial material that passed a 40-mesh screen was reduced from 100 to 50 percent. Finely divided materials were found to granulate easily, regardless of whether they were soluble or insoluble. As a rule, the presence of a soluble salt in a mixture tended to increase the crushing strength of the granule. Some easily soluble, readily fusible materials, such as urea and ammonium nitrate, were particularly effective in promoting granulation because of the cohesiveness they exhibited.

A variation of the rotary-drum granulating method, described in last year's report, was developed, wherein the desired moisture content and temperature were attained by the addition of steam to the mixture during the granulating step. The heat, necessary to facilitate granulation, may also be supplied by ammoniating the mixture during the granulation process, the heat developed in this way being sufficient in some instances to elevate the temperature to 100° C. and evaporate some of the moisture present.

In connection with the investigations to determine the effect of fertilizer mixtures and salts on the concentration of the soil solution and thus obtain information on the burning effects of the fertilizers on plants, it was found that when single- and double-strength fertilizers containing similar salts were applied at equivalent rates to the soils employed (Norfolk sandy loam and Cecil clay loam) the double-strength fertilizer had less effect than the single-strength materials in raising the concentration of the soil solution. However, when two double-strength mixtures containing dissimilar salts were applied, the mixture containing more soluble salt had the greater effect on the concentration of the soil solution. A 6-8-4 mixture prepared according to present manufacturing practice was found to have less effect than the 3-8-4 mixtures formerly used. The results indicate that from the point of view of injury to the crop through burning, it is no longer necessary to apply nitrogen to cotton in split applications. When granulated and powdered fertilizers of identical composition were applied to Norfolk sandy loam and Cecil clay loam, the soil solutions from the soils treated with the granulated mixtures had slightly lower concentrations than those from the soils treated with the powdered mixtures.

Determinations were made of the heats of reaction between a superphosphate (made from a Tennessee phosphate rock) and various quantities of anhydrous ammonia ranging from 1 to 10 percent, of the specific heats of a number of fertilizer materials, and of the heat of formation of monocalcium phosphate, for the purpose of ascertaining the maximum temperature rise that might occur in the ammoniation of various fertilizer mixtures. The heats of reaction found by experiment were in good agreement with the calculated values for the heats of reaction in the ammoniation of superphosphate and superphosphate mixtures. Confirmation was thus obtained of the reactions that were assumed to take place during ammoniation.

In further studies to determine the cause of reversion of the phosphoric acid in commercial ammoniated fertilizer mixtures that contain dolomite, a series of synthetic ammoniated mixtures were prepared, from which one or more of the impurities normally occurring in commercial superphosphate were eliminated in turn. It was found that reversion occurred in fluorine-free mixtures as well as in those that contained fluorine.

Fertilizer samples, representative of the mixed fertilizers consumed in the United States, were analyzed for all the common elements. Work is in progress on the determination of the trace elements in these samples.

The substitution of calcium metaphosphate for ordinary or double superphosphate in a fertilizer mixture was found to cause increased hygroscopicity of the mixtures. Mixtures containing crude calcium metaphosphate caked badly. Admixtures of calcined phosphate to salt pairs did not increase their hygroscopicity and did not have a retarding effect on the absorption of water by urea. Granulated urea was found to absorb moisture at about the same rate as does crystalline urea.

## SOIL CHEMISTRY AND PHYSICS RESEARCH

### SELENIUM IN SOILS

Further investigations were made on the occurrence of selenium in soils. Highly seleniferous areas were found in New Mexico and Colorado. A detailed examination of the soils in southeastern Colorado revealed the existence of a soil area in excess of 3,000 square miles, which is capable of producing vegetation toxic to animals. A similar but less extensive area was discovered in northeastern New Mexico. Investigation of the occurrence of selenium in semiarid areas was continued southward into Mexico during the winter. Domestic-animal and human symptoms indicated the presence of highly seleniferous areas in certain parts of Mexico.

Observations on the plants growing in seleniferous areas led to the inference that the occurrence of selenium in the soil influences the relative abundance of different plant species and perhaps even determines the presence of certain species.

Evidence was obtained that irrigated seleniferous soils produce vegetation of much lower selenium content than similar nonirrigated areas, provided sufficient selenium-free water is used.

A study of many soil profiles in seleniferous areas showed no constant relation in the distribution of the selenium within the soil profile and no constant relation between the selenium in any part of the soil profile and that of vegetation produced on it.

In connection with the identification of injuriously seleniferous areas, it has been necessary to ascertain whether the poisonous effects of selenium compounds on vegetation are influenced by the character of the soil. Sodium selenate is highly toxic to many crop plants, although a few plants are rather tolerant of it. In pot experiments  $1\frac{1}{2}$  parts of selenium as sodium selenate in a million parts of soil were found sufficient to reduce the growth of millet by 50 percent. Toxicity of the selenate varied little in soils of widely different character with regard to soil colloids, but it was less in those of high soluble sulphate content than in those of low sulphate content. Preliminary studies on sodium selenite, another form in which selenium occurs in soils, indicated that its behavior would be decidedly different from that of the selenate.

Data obtained by this Bureau in its selenium investigations have been employed by the Resettlement Administration in efforts to determine the most satisfactory uses for extensive seleniferous lands acquired by that organization in South Dakota.

### NONFERTILE SOILS

Soils from the vicinity of barite areas in four States were found to contain from 0.1 to 3.69 percent of barium calculated as the oxide. A high percentage of barium apparently does not cause soil to be nonfertile when a sufficient quantity of lime is present, but it may be one of the causes of infertility in soils of low lime content. The maximum quantity of barium oxide found in plants grown on barium-containing soils was 0.15 percent.

Analyses of the leaves of trees growing on soils of different composition showed that the ash composition of the leaves and the composition of the soil are dependent on each other. Black locust leaves growing on limestone soil contained three times as much lime as the same kind of leaves growing on a lime-poor Podzol soil. The quantity of alumina and manganese found in leaves depends mainly on the soil reaction and not on the quantities of these elements in the soil, except when they are present in limiting quantities.

Certain leaves when grown on acid soils have been found to contain such a high percentage of alumina as to greatly affect the distribution of alumina in the soil profile. In pure stands of hickory and sweetleaf, the leaves of which contain, respectively, 1.5 and 4.5 percent of alumina, as much as 100 pounds of alumina per acre may be brought up from within the soil and deposited on its surface each year. The quantity of manganese in the leaves of trees grown

in very acid soils reaches a maximum of nearly 0.5 percent and thus becomes a major element in the ash composition of the leaves. The distribution of manganese in the soil profile must be affected to a marked extent by such a high percentage in the leaf litter.

Leaves of trees growing on soils of low lime content may be so low in lime as to form an acid litter which decomposes slowly and forms a mat that persists and accumulates, resulting in Podzol soils when climatic conditions are favorable. Hemlock needles and pine needles are particularly low in lime, and for this reason and perhaps others, hasten the podzolizing action as compared to the leaves of hardwood trees.

#### POORLY DRAINED SOILS

A study of soils formed under different conditions of drainage on the Atlantic Coastal Plain showed that the chemical composition of the colloids in poorly drained soils is fairly constant, approximating very closely the theoretical composition of halloysitic acid ( $3\text{H}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ ). The colloids of all the coastal plain soils were low in bases but were lowest in the most poorly drained soils. The amount of eluviation was found to increase with the degree of drainage.

#### ALLUVIAL SOILS

A field study was made and samples collected of alluvial soils from the bottom lands of the following rivers: Mississippi (lower, middle, and upper), Black, Ohio, Tennessee, Cumberland, Duck, and Clinch. Since these soils show but slight profile development, they were sampled at such depths as to obtain material most representative of assumed A, B, and C horizons. At one place in the middle Mississippi area, 11 soil samples were obtained at various depths, ranging from the surface of the ground to the depth of the coastal plain material—100 feet. Fourteen samples of sedimentary material deposited by the January 1937 flood of the Ohio River were obtained for study.

#### LIMESTONE SOILS

Investigation of the limestone soils of the Great Valley was continued with a study of Lebanon silt loam from Phelps County, Mo., and of Hagerstown silt loam from St. Francois County, Mo. These soils, all derived from residual material from the decomposition of limestone, range from the Hagerstown which is typically a Gray-Brown Podzolic soil, through intermediate soils to the red soils of the Decatur series. The major variations in composition of the colloids involve the following elements: Iron, manganese, phosphorus, and the bases. Silica and alumina do not vary through a wide range.

#### GRANITIC SOILS

Samples of profiles of soils from New England and the Piedmont, derived from granitic material, were collected and studied with respect to composition and the chemical and physical properties of their colloids. The results of this study showed that the podzolization process has kept pace with the disintegration of rock and has been extremely active in the formation of colloids of the Gray-Brown Podzolic soils derived from glacial drift. Podzolization is slight in the same group of soils developed on the comparatively ancient formations. The dominant soil-forming process of each group is hydrolysis: it is influenced by the parent material and is intensified by an increase in the mean annual temperature. Environmental conditions tend to produce, in the soils of New England and the Piedmont, colloids of a wide range in composition. Parent material, temperature, rainfall, and vegetation are the primary factors affecting the composition of the colloids.

#### MOISTURE IN SOILS

For studying the relative moisture-retaining capacities of different soils and the problem of why and how some soils hold more moisture than others, determinations of the exact temperatures at which soils freeze and thaw and the quantities of heat released or absorbed in these changes of state have been found very useful. Measurement of the changes in dielectric constant of soils was found to be an accurate means for obtaining data relative to their freezing and thawing. During the year an apparatus was designed and constructed for this purpose. Results obtained by the use of this apparatus showed that in the lower range of moisture content, at least, soils begin to freeze and are

completely thawed at the same temperature, indicating no wetting and drying hysteresis at this temperature.

The same apparatus and method were found to be useful in determining the temperature and time required to freeze vegetables and fruits and other materials.

#### ADSORPTION OF GASES BY SOILS

In order to gain further insight into the surface relationships in soils, a study is being made of the behavior of soils and their colloids with respect to adsorption of gases. Special apparatus for this purpose was constructed with the aid of P. H. Emmett of the Fertilizer Research Division. Experimental data have been obtained on the adsorption of nitrogen at 0° and -183° C., of oxygen at 0°, and of carbon dioxide at 0°, by soil and colloid samples of single horizons representative of five great soil groups. The adsorption values found for the soils were used to calculate the percentage of colloidal material present, and the calculated values agreed reasonably well with those obtained by mechanical analysis. An attempt is being made to use the adsorption isotherm for nitrogen at -183° C., to estimate surface areas and to correlate the magnitudes thus obtained with known chemical and physical properties of soils and soil colloids. The magnitude of the adsorption of individual gases differs for soils of various great soil groups and is lower for the soils than for their corresponding colloids. Each soil and colloid has a characteristic adsorption value which is correlated with the general classification of soils.

The density of the various soil materials used in this work was obtained by a new modification of the usual pycnometer method. Helium was used instead of a liquid to fill the pycnometer space, thus assuring a complete penetration of the pores of the solid and eliminating the possibility of any chemical or physical reactions.

#### STRUCTURE OF PEAT

The colloidal material in the organic matter of peats is being studied, with particular reference to its dispersibility and the base combining capacity of its acids.

The segregation of colloidal peat acids by a purely mechanical dispersion procedure, such as is used in the case of mineral soils, was not found possible. Even the use of such drastic hydraulic agitation as given by a colloid mill proved inadequate. A system of fractionation by chemical means was employed for the separation of colloidal and other acids. This consisted of a preliminary electrolysis followed by treatment with 1-percent solution of sodium hydroxide at 70° to 75° C. The material dispersed to colloidal size as well as that dissolved by the alkaline treatment was separated by means of a supercentrifuge and subsequent filtration.

A method of electrometric titration of peats and peat fractions was developed. In this method a fairly definite end point was consistently indicated by breaks in the titration curve at pH values of approximately 7.2 to 8.8. The success of this method of titration is based on the use of a very dilute suspension and a time reaction of from 8 to 10 days before making pH measurements. The peat fractions differed widely with respect to the relative amounts of base required for neutralization. As much as 70 percent of the total acidity of the original peat was found in the so-called humic-acid fraction which was the material dissolved by the alkaline solution and precipitated by acid. The base-combining capacities of a number of entire peat samples were found to vary from approximately 2.5 to 3.5 milliequivalents of barium per gram of peat.

#### QUICK CHEMICAL TESTS FOR SOILS

Comparative results have been obtained by various quick tests on widely divergent soils. These tests are believed to be of very limited usefulness in the hands of an untrained person, except as he may wish to conduct local soil experiments. They are of greater value to agronomists who have good background of supplemental information. Chemical tests may be of marked value to one who has knowledge concerning the general level of chemical values obtained for soils of some particular series, their productivity with various fertilizer treatments, and their suitability for particular crops, and they may be very helpful for the detection of overfertilization in special instances, as with greenhouse soils.

### SOIL REACTION

During the last 11 years a large number of hydrogen-ion concentration determinations have been made on soils from all parts of the United States, including samples of profiles from all the major soil groups and most of the more important soil series. Work has been started on the organization of these voluminous data into form for publication. The data are adequate to show, in general, the maximum and minimum pH values, as well as the predominating values characteristic of the various soil groups and soil series of this country.

### SERVICE WORK

Chemical and mechanical analyses of soils, as well as certain other physical tests including the determination of moisture equivalent, are made in considerable numbers. During the last year, the service work included 145 complete chemical analyses of soils and soil colloids, 1,331 nitrogen determinations, and 732 mechanical analyses of soils. Many of the mechanical analyses were made for the Soil Survey Division and for the Federal Emergency Administration of Public Works, and numerous miscellaneous tests were made for the Soil Conservation Service.

### SOIL SURVEY

The work of the Soil Survey Division comprises the determination of the character of soils, definition of soil types, development of a uniform system of soil classification, delineation upon maps of the boundaries of each soil type, and the interpretation of the capabilities of the soils for the production of crops, grasses, and trees under different kinds of management. The ultimate purpose of this research is to provide accurate soil maps of the country which are necessary for the classification of rural lands, and for the factual basis in the development of rational programs of land use, consistent with conservation and the welfare of the agricultural people, whether planned by public agencies or individual farmers.

During the year, 27,350 square miles of rural lands in 33 States and Hawaii were mapped by the Soil Survey Division. This brings the total area covered by the soil survey to more than one-half of the arable lands of the Nation. The soil maps, together with the accompanying reports giving descriptions of the soils and their uses, provide farmers, research workers, extension leaders, and local officials with a practical working handbook of the land for the area covered. In order that the results of experiments on farm land may be utilized in connection with problems of land use, it is necessary that information be given regarding the type of soil on which the experiments are made. Once an accurate map of the soil types is made, the results may be extended to individual farm areas having the same soil types. The continued extension in the use of soil maps, wherever they are available, for determining land-use policies testify to their accuracy and value.

### STATE COOPERATION

All this research is accomplished in direct cooperation with local agencies, especially the State agricultural experiment stations. In this way both the broader perspective of the Federal organization and the more detailed local knowledge of the State agencies are utilized to the best advantage, each supplementing the other. For example, the Bureau is cooperating with the seven States having responsibilities for the programs of agricultural development in the watershed of the Tennessee River. The agricultural experiment stations of these States also are cooperating with the Tennessee Valley Authority in expediting the completion of the detailed soil survey of that area and in conducting research on the various soil types in order to determine their capabilities for agricultural use. Accurate detailed maps showing the location of the various soil types having particular possibilities for use under different types of management are essential in developing systems of agriculture, offering security to the farm family, and for improvement of the land, prevention of erosion, and the protection of streams and reservoirs.

Similarly, the Bureau is cooperating with a great many other States in order to obtain the information essential to a solution of their land-use problems. In several of the Western States, such as California, Oregon, Utah, Washington, and others, information on soils is essential for planning the extension of certain special crops and for the proper planning or extension

of irrigation, in order to avoid areas subject to an accumulation of alkali or those having excessive water requirements or other draw-backs.

The problems in each State, and even in each county, are more or less individualistic and require somewhat different approach for their solution. This fact emphasizes the necessity of cooperation among Federal, State, and local people in order that a balanced point of view may be obtained. The soil maps are made in such a way as to indicate those physical differences in the land which determine or influence its capabilities for use. Usually an area has more than one problem; the soil map must carry the physical information pertinent to them all. Thus, from the soil map, other relatively simple maps, showing only one characteristic, may be made by interpretation. For example, supplemental maps showing only erodibility, lime requirement, drainage, or similar features can be made from the soil map. Others may be prepared, showing relatively simple groups of soils according to their adaptability to alfalfa, tree fruits, or other crops or groups of crops. Using the soil map as the base, other maps may be prepared, showing the general suitability of the land for agricultural or other uses as a means for developing plans for rural zoning. All these relatively simple maps would differ from one another, although each would be taken from the master soil map. In North Dakota, for example, soil maps are used as the basis for rating rural land for tax assessments.

#### REGIONAL INVESTIGATIONS

Since it is of the utmost importance that the nomenclature of soil types, their definitions, and classification be everywhere consistent, regional comparisons of soil types are essential to the general program. These activities are conducted by the supervisory scientists of the Bureau, who are responsible also for the nomenclature used on the detailed soil maps. During the year special regional studies have been made in the western part of the country, where the present information is inadequate for the proper conduct of more detailed surveys.

#### PRODUCTIVITY RATING

A system is being developed rapidly for relating the results of experience and experiments to the individual soil types (and phases), in order that each soil shown on the map may be given a definite rating according to its productivity and crop adaptability. Such a system involves consideration of the inherent productivity of the soil as well as its productivity under different systems of management. Many of the soil surveys being published currently contain tables showing these productivity ratings, and it is expected that this research will be pushed to the point where such tables will accompany all soil surveys as soon as the system is broadened and fully developed.

#### PREPARATION OF SOIL MAPS

After the soil maps have been drawn in the field and the necessary description of the soils and their capabilities for use prepared, the maps are redrawn for publication, and the reports, including soil descriptions, recommendations for their use, agricultural statistics and similar matters, are printed. These reports and maps generally are published in county units, although occasionally the unit of publication is more or less than one county, such as a stream valley or a watershed. When completed the reports and maps are made available to the public and to all Government agencies having need for them.

The progress of soil mapping is shown in tables 1 and 2, and a list of soil surveys published during the last year is included with the list of publications.

TABLE 1.—*Individual areas surveyed and mapped during the fiscal year ended June 30, 1937*

State or Territory	Area	Area surveyed	
		Square miles	Acres
Alabama.....	{ Lee County.....	1 188	120, 320
	{ Macon County.....	1 478	305, 920
Arizona.....	{ Yuma-Gila area.....	114	72, 960
California.....	{ Bakersfield area.....	1, 120	716, 800
Colorado.....	{ Kings County.....	290	185, 600
	{ Washington County.....	1 605	387, 200

<sup>1</sup> These figures do not include portions of these areas surveyed in preceding years.

TABLE 1.—Individual areas surveyed and mapped during the fiscal year ended June 30, 1937—Continued

State or Territory	Area	Area surveyed	
		Square miles	Acres
Florida	Alachua County	229	146,560
Georgia	Catoosa County	169	108,160
	Hall County	159	37,760
Hawaii	Hawaiian Islands	11,969	1,260,160
Idaho	Bingham County	1249	159,360
	Brown County	1272	174,080
	Fulton County	47	30,080
Indiana	Johnson County	41	26,240
	Martin County	339	216,960
	Morgan County	27	17,280
	Allamakee County	1154	98,560
	Jackson County	1104	66,560
Iowa	Story County	1183	117,120
	Tama County	318	203,520
	Calloway County	1170	108,800
Kentucky	York County	278	177,920
Maine	Clinton County	1324	207,360
	Mason County	1220	140,800
Minnesota	Newaygo County	30	19,200
	Washington County	153	97,920
Mississippi	Tishomingo County	1225	144,000
	Cass County	1302	193,280
Nebraska	Cherry County	1908	581,120
	Lancaster County	194	60,160
	Otoe County	36	23,040
	Cheshire County	400	256,000
New Hampshire	Coos County	1,053	673,920
	Sullivan County	1277	177,280
	Albany-Schenectady Counties	148	30,720
New York	Niagara County	1178	113,920
	Sullivan County	232	148,480
	Henderson County	175	48,000
	Jackson County	43	27,520
North Carolina	Madison County	1189	120,960
	Swain County	343	219,520
	Transylvania County	169	108,160
	Warren County	1115	73,600
North Dakota	Morton County	1317	202,880
	Williams County	209	133,760
Ohio	Tuscarawas County	1132	84,480
	Choctaw County	1487	311,680
Oklahoma	Creek County	1170	108,800
	Woods County	399	255,360
	Baker County	27	17,280
Oregon	Umatilla area	1256	163,840
	Bucks County	169	44,160
Pennsylvania	Crawford County	221	141,440
	Fayette County	36	23,040
	Newport-Bristol Counties	132	20,480
Rhode Island	Providence County	199	127,360
South Carolina	Pickens County	1172	110,080
	Bedford County	35	22,400
	Cumberland County	253	161,920
Tennessee	Hamilton County	1268	171,520
	Humphreys County	1107	68,480
	Lincoln County	1291	186,240
	Roane County	1123	78,720
	Brown County	190	57,600
Texas	Dimmit County	369	236,160
	Fannin County	1236	151,040
Utah	Provo-Lehi area	119	76,160
	Isle of Wight County	198	62,720
	Princess Anne County	56	35,840
	Russell County	1407	260,480
Virginia	Smyth County	89	56,960
	Tazewell County	107	68,480
	Washington County	1280	179,200
	Clallam County	53	33,920
	King County	33	21,120
Washington	Kittitas County	1195	124,800
	Snohomish County	1545	348,800
	Yakima County	1180	115,200
	Greenbrier County	1431	275,840
Wyoming	Campbell County	12,205	1,411,200
Total		22,113	14,152,320

<sup>1</sup> These figures do not include portions of these areas surveyed in preceding years.

TABLE 2.—Areas surveyed and mapped in the several States during the fiscal year ended June 30, 1937, and the areas previously reported

## DETAILED

State or Territory	Work dur-	Work previ-	Total	
	ing 1937	ously reported	Square miles	Acres
Alabama.....	666	59,836	60,502	38,721,280
Arizona.....	114	4,955	5,069	3,244,160
Arkansas.....	15,547	15,547	15,547	9,950,080
California.....	1,410	38,562	39,972	25,582,080
Colorado.....	605	6,128	6,733	4,309,120
Connecticut.....	1,704	1,704	1,704	1,090,560
Delaware.....	2,276	2,276	2,276	1,456,640
Florida.....	229	15,160	15,389	9,848,960
Georgia.....	228	36,452	36,680	23,475,200
Hawaii.....	1,969	699	2,668	1,707,520
Idaho.....	249	12,716	12,965	8,297,600
Illinois.....	726	6,770	6,770	4,332,800
Indiana.....	726	21,645	22,371	14,317,440
Iowa.....	759	51,445	52,204	33,410,560
Kansas.....	170	16,854	16,854	10,786,560
Kentucky.....	170	5,784	5,954	3,810,560
Louisiana.....	278	17,431	17,431	11,155,840
Maine.....	278	2,197	2,475	1,584,000
Maryland.....	13,959	13,959	13,959	8,933,760
Massachusetts.....	574	8,811	8,811	5,639,040
Michigan.....	153	32,693	33,267	21,290,880
Minnesota.....	225	12,867	13,020	8,332,800
Mississippi.....	225	30,943	31,168	19,947,520
Missouri.....	37,177	37,177	37,177	23,793,280
Montana.....	1,340	3,287	3,287	2,103,680
Nebraska.....	1,340	69,108	70,448	45,086,720
Nevada.....	1,730	652	652	417,280
New Hampshire.....	1,730	3,390	5,120	3,276,800
New Jersey.....	458	9,895	9,895	6,332,800
New Mexico.....	934	2,565	2,565	1,641,600
New York.....	458	37,535	37,993	24,315,520
North Carolina.....	934	48,809	49,743	31,835,520
North Dakota.....	526	22,523	23,049	14,751,360
Ohio.....	132	18,650	18,782	12,020,480
Oklahoma.....	1,056	25,283	26,339	16,856,960
Oregon.....	283	15,991	16,274	10,415,360
Pennsylvania.....	326	22,689	23,015	14,729,600
Puerto Rico.....	3,765	3,765	3,765	2,409,600
Rhode Island.....	231	1,690	1,921	1,229,440
South Carolina.....	172	27,082	27,254	17,442,560
South Dakota.....	1,077	8,286	8,286	5,303,040
Tennessee.....	1,077	12,687	13,764	8,808,960
Texas.....	695	66,730	67,425	43,152,000
Utah.....	119	3,096	3,215	2,057,600
Vermont.....	1,037	1,175	1,175	752,000
Virginia.....	1,037	15,498	16,535	10,582,400
Washington.....	1,006	12,388	13,394	8,572,160
West Virginia.....	431	23,904	24,335	15,574,400
Wisconsin.....	2,205	26,659	26,659	17,061,760
Wyoming.....	2,205	12,582	14,787	9,463,680
Total.....	22,113	948,530	970,643	621,211,520

## RECONNAISSANCE

Alaska.....		31,915	31,915	20,425,600
Arkansas-Missouri.....		58,000	58,000	37,120,000
California.....		32,135	32,135	20,566,400
Kansas.....		39,960	39,960	25,574,400
Michigan.....		1,322	1,322	846,080
Minnesota.....	433	12,311	12,744	8,156,160
Montana.....	2,935	51,943	54,878	35,121,920
Nebraska.....		53,064	53,064	33,960,960
North Dakota.....		39,240	39,240	25,113,600
Ohio.....		41,420	41,420	26,508,800
Pennsylvania.....		41,405	41,405	26,499,200
South Dakota.....		41,400	41,400	26,496,000
Texas.....		152,855	152,855	97,827,200
Vermont.....		9,124	9,124	5,839,360
Washington.....	1,869	16,540	18,409	11,781,760
Wisconsin.....		14,425	14,425	9,232,000
Total.....	5,237	637,059	642,296	411,069,440



## SPECIAL WORK OF THE SOIL SURVEY

For the successful prosecution of many of the activities of the Federal Government and cooperating State agencies it has been necessary for scientists in the Soil Survey Division to interpret the available soil data in terms of the particular objective at hand. As the soil-survey data are fundamental and basic for almost all kinds of agricultural activities, thousands of private individuals and organizations, as well as public agencies, seek the advice and assistance of soil scientists of the Bureau as to the location of soil types adaptable to various crops and how the various soil types may be managed. Requests of this kind have greatly increased in the last few years.

For many years scientists in the Soil Survey Division have called attention to the serious injury, through erosion, to soils when they are improperly used. The fundamental nature of the soil type determines its erodibility under different methods of management. The soil maps serve as a basis for erosion surveys and erosion-control projects. In many instances where these maps have been unavailable, soil scientists of this Bureau have assisted in making special studies and maps for the use of Federal and State agencies charged with the responsibilities for the control of erosion. Scientists of the Bureau are assisting the Tennessee Valley Authority, Soil Conservation Service, and other agencies toward this end.

In addition to the attention given to the possibilities of land for irrigation in connection with the regular soil-survey projects, special reports have been prepared in regard to the suitability of land for irrigation in areas under the jurisdiction of other governmental agencies.

Work has been continued in the investigation of peat lands in cooperation with other Federal and State agencies, particularly from the point of view of the relationship of such soils to the conservation of land and prevention of floods. A special report describing the characteristics and distribution of various kinds of organic soils and peat in the Pacific Coast States has been published. Special studies and reports have been made of peat areas in connection with the work of the Bureau of Biological Survey and other agencies having special problems in the utilization of these lands.

The Soil Survey Division has continued to assist in the selenium investigations being carried on by the Department. Examinations of soils and vegetation in several States, where excess selenium in the soil is an important factor in their utilization, have been made. The results of these researches are incorporated in reports published by the Bureau.

Land appraisers of the Farm Credit Administration and other agencies have made wise use of the soil-survey maps and reports. Through personal contact and in other ways, scientists on the Soil Survey Division staff have assisted in the interpretation of soil data as they bear on land appraisal of rural lands affected by flood-control projects.

## INFORMATION AND PUBLICATIONS

During the year, Department publications from this Bureau were 26 soil-survey reports, 7 technical bulletins, 1 circular, 3 miscellaneous publications, 3 articles in the *Journal of Agricultural Research*, 15 mimeographed publications, 205 articles in outside publications, and 14 patents. In cooperation with the press and radio services, the Bureau furnished information on various phases of its research.

The following lists show the publications and patents from the nine research divisions of this Bureau:

## LIST OF PUBLICATIONS

## CARBOHYDRATE RESEARCH DIVISION

Apparent and true solids of sugarcane juice—relation of difference to ash content. By C. A. Fort and N. McKaig, Jr. *Indus. and Engin. Chem., Analyt. Ed.* 8: 333-335. 1936. (With Bur. Plant Indus.)

Report of studies on uniformity of quality of sugars. I. Chemical studies of beet sugars collected during 1935 campaign. By J. C. Keane, S. Byall, and J. A. Ambler. 17 pp. 1936. [Mimeographed.]

Report of studies on uniformity of quality of sugars. I. Chemical studies of beet sugars collected during 1936 campaign. By J. C. Keane, S. Byall, and R. M. Kingsbury. 19 pp. 1937. [Mimeographed.]

Impurities in white sugars. IX. Loss of anions from certain salts during evaporation of concentrated sugar solutions. By J. A. Ambler. *Indus. and Engin. Chem.* 28: 1266-1268. 1936.

Recent advances in the technology of beet sugar manufacture. By J. C. Keane and

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- Yield and quality of table sirup from fresh cut vs. old-cut sugarcane. By C. F. Walton, Jr., and C. A. Fort. Sugar Bull. 14 (22): 6-7. 1936.
- Quality of table sirup from the newer sugarcane varieties. By C. F. Walton, Jr., and C. A. Fort. Sugar Bull. 15 (3): 4-6. 1936.
- Food plants of the North American Indians. By E. Yanovsky. U. S. Dept. Agr. Misc. Pub. 237.
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- Sweetpotato starch—a new farm industry for the south. By H. G. Knight. Manfr. Rec. 56 (2): 40-41 and 68. 1937.
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- Composition of the oil of the American black walnut. By G. S. Jamieson and R. S. McKinney. Oil and Soap 13 (8): 202. 1936.
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## SOIL SURVEYS

- Alabama*
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Mobile County.  
Winston County.
- California*
- Alturas area.  
Lodi area.
- Indiana*
- Randolph County.  
Rush County.
- Kentucky*
- Fayette County.
- Michigan*
- Iron County.
- Nebraska*
- Greeley County.  
Rock County.
- New Mexico*
- Roswell area.
- New York*
- Broome County.  
Rensselaer County.
- North Carolina*
- Lee County.  
Washington County.
- Ohio*
- Brown County.  
Putnam County.
- Pennsylvania*
- Indiana County.
- Texas*
- Cass County.  
Falls County.  
Hardeman County.  
Wheeler County.
- Vermont*
- Reconnaissance of State of Vermont.
- Virginia*
- Augusta County.
- West Virginia*
- Randolph County.
- MISCELLANEOUS
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