American Ergot.—The utilization of American ergot, obtained from rye screenings, has been under consideration in coöperation with the Central Inspection District located in Chicago. The products examined were in excellent condition and were found to be quite active physiologically; difficulties, however, are encountered in isolating the ergot on an efficient commercial scale from the creenings.

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# II.

# PHARMACOGNOSY INVESTIGATIONS.

Studies in Brassicas.—Growing experiments and chemical studies were carried out with several mustards and mustard substitutes.(1) The seeds were obtained from different parts of the world, and especially from Japan, China and India. They were planted in Arlington, Va., Yarrow, Md., and Urbana, Ill. The plants grew well in all three places, but especially vigorously in the rich soil of Illinois. The amount of seeds obtained per plant, and the pungency of the seeds, were great. In a number of instances sufficient material from individual plants was secured for the determination of the latitude in the amount of glucoside present. The amount of volatile oil found reached in certain cases as high as or even higher than any reported in literature. While the growing experiments were only carried out on a small scale, the results suggest that the commercial cultivation of all the different varieties, and especially the Japanese and white mustard, should be successful.

Preliminary experiments concerning the distribution of the glucoside or glucosides within the plant indicate that the fresh tissue of the different parts of the entire plant, roots, stems, leaves and fruitpods, yield an oil with characters similar to that obtained from the seeds. The enzyme, effecting the hydrolysis of the glucoside, appears present in the green plant in a highly active form, since the glucoside in the growing tissue is far more readily hydrolyzed than in the seeds.

A monograph on Chinese Colza is being published as a Department Bulletin.(2) It discusses the chemical, anatomical data of the seed, and the physiological data of the seed and seed constituents, and the morphological characters of the plant.

in the different stages of its growth. A brief note has been prepared for a trade journal, discussing especially the value of the seed as an oil seed and as a substitute for rape.(3) While the seed had been first offered to the trade as mustard, the investigation proved the distinct difference from mustards and the close relationship to the colza group.

Studies on Oxalic Acid in Foods and Spices.—Studies on this subject are nearing completion. They had the object to determine the presence of oxalic acid as calcium oxalate or otherwise in the plant tissue by means of microscope and microchemical reagents;(4) and determine also quantitatively the amount of oxalic acid in different foods and spices.(5) Interesting data have been obtained which should be helpful to food and drug analysts, as well as to physiologists or pharmacologists.

Evidence that oxalic acid sublimes already at  $100^{\circ}$  C. and below was secured, and a new microchemical reaction with resorcin-sulphuric acid was worked out.

Studies of Spanish Digitalis, Digitalis thapsi.—In continuation of previous studies, incident to the identification of this spurious Digitalis (see S. R. A 19)(6) the plants grown from the seeds, that had been separated from the material imported, were harvested, being in their second and third year of growth. Further chemical, microchemical and pharmacological work is planned.

Studies of Saponins.—The work on saponins and saponin-yielding plants was carried out jointly with Drs. Johns and Chernoff in continuation of previous studies(7)–(10) on the same subject. The recent studies were concerned with plants growing quite abundantly in American deserts and are now partly utilized as emergency feed for cattle, as soap substitute and as fiber material. Manuscripts on Agave lechuguilla, Yucca glauca (angustofolia) and Yucca radiosa (elata), the latter usually referred to as soap weed, have been prepared for publication. Special attention was paid to the distribution of the saponins within the plant, to their chemical and hemolytic properties and probable function in the metabolism of the plant. The observation that in these desert plants the saponins occur in the cell sap or veins suggest that they are not only used as food material, suggested by the presence of one or more sugar molecules in saponins, but also serve, since they are generally hygroscopic, to retain moisture within the tissue during long periods of draught.

Cyanogenesis.—1. Studies of Edible and Poisonous Varieties of Beans of the Lima Type (Phaseolus lunatus).—These studies,(11) nearing completion, had the object in mind to establish botanical and chemical characteristics of seeds and plants, to find or develop reliable methods for testing and estimating the glucoside, as well as the hydrocyanic acid obtained from it by either acid or enzymatic hydrolysis,(12) to determine the latitude of varietal and individual variation in the glucosidal content and to render the beans harmless by cultural, chemical or physical means. Of the results obtained it may be stated that the seeds, and the plants in different stages of growth can be quite readily distinguished from other Phaseolus species of economical value in this country. The species Phaseolus lunatus is also chemically distinct from other Phaseolus species by lacking calcium oxalate in the seed coat and containing in all the varieties examined the cyanogenetic glucoside. Parts containing this, such as the inner kernel or the remainder of the

seed placenta, often visible at the hilum, are stained intensely yellow upon exposure to ammonia. This test, together with the characteristic arrangement of the veins in the seed coat, radiating from the hilum to the dorsal venture, are means of differentiation which can be readily determined macroscopically, while the lack of calcium oxalate can be easily established microscopically. The American grown varieties "Limas" appear to contain the cyanogenetic glucoside in the smallest amount of any grown anywhere, and the beans may, therefore, as far as the results at hand show, be considered harmless.

2. Fate and Significance of Linamarin in the Metabolism of the Bean Plant, Flax, Etc.—To solve this problem seeds of several varieties of *Phaseolus lunatus* and also flax seeds have been germinated and the amount of HCN present under different conditions of germination has been determined. These studies are being continued with plants in different stages of growth and grown under different conditions.

The effect of frost and drying has also been under consideration. A loss of HCN in the first case is certain, and in the second case strongly indicated. No free HCN has positively been found in the plant and no HCN is apparently exhaled under normal conditions. The glucoside in the leaves and other growing tissue is evidently much more speedily hydrolyzed than in the seeds, probably due to the fact that the enzyme, effecting the hydrolysis of the glucoside, is in a far more active form present in the green growing tissue than in the seed.

3. Distribution and Occurrence of Cyanogenetic Glucosides.—In continuation of previous studies(13) other vegetable products were tested for hydrocyanic acid, however with negative results. The following may be mentioned: Horse beans (Vicia faba), Tepary beans (Phaseolus acutifolius var. latifolia), Azuki beans (Phaseolus angularis), a Japanese or Chinese bean, also called "Adzuki," Jack beans (Canavalia ensiformis), Velvet beans (Stizolobium deeringianum), Spanish or Runner beans (Phaseolus multiflorus), Soja beans (Soja hispida), and Mexican and oriental (Japanese, Chinese and Manchurian), varieties of common beans (Phaseolus vulgaris).

4. Isolation of Hydrocyanic Acid and the Glucoside Yielding it from Indian Beans of Lima Type, Phaseolus lunatus, from Flax, Linum usitatissimum, Etc.—The studies have resulted in a very simple method of isolation of the glucoside from the beans. The ground bean meal is extracted repeatedly with ethyl acetate, and this then shaken out several times with very small amounts of water. Upon evaporation of the water, preferably without application of heat, the glucoside is obtained in surprisingly pure, white to yellowish crystals. For the isolation of the glucoside from flax this method had to be modified, since in flaxseed the great amount of oil, of water-soluble slime and of wax, interfered very much.

Preliminary experiments with certain chemicals acting upon the glucoside or the beans effecting a transformation of the poisonous glucoside have been very successful and will be continued on a larger scale and extended to other cyanogenetic products.

Study of Piper bredemeyeri, an Adulterant of Matico.—An examination of the volatile oil revealed the absence of asaron, found in matico, Piper angustifolium, and of matico camphor, found in Piper angustifolium var. ossanum. Over 50 percent of dill apiol were present in the volatile oil of P. bredemeyeri; in this and

other respects the oil resembled the volatile oil of *Piper mandoni*, as reported in literature. The chemical and botanical similarities of *P. bredemeyeri* and *P. mandoni* suggest that the name *P. mandoni* has been given to plants belonging to the species *P. bredemeyeri*.(14) Recent reports indicate that material of matico and especially also of volatile matico oil vary greatly in composition. The medicinal qualities of matico have been questioned. Matico has been deleted from the U. S. Pharmacopoeia, though it is now included in the National Formulary. While the medicinal value, as said, of matico is in question, that of the adulterant *P. bredemeyeri* and other related Piper species is still more in doubt. No physiological tests have as yet been made. Attempts to secure further authentic material of different Piper species were unsuccessful.

Studies of Cedron Seed, Simaba cedron, and the Glucoside Cedrin.—From cedron seed, originating in Central America, and used there against snake bites and yellow fever, the bitter principle "cedrin" had been isolated some time ago.(15) Certain physical and chemical properties had been determined and the glucosidic character definitely established. The nature of the sugar in the glucoside could, however, not be ascertained due to insufficient amounts of cedrin available. Recently, however, after prolonged attempts, more material of cedron seeds was obtained through the efforts of the Office of Foreign Seed and Plant Introductions, B. P. I., U. S. Department of Agriculture. The studies can therefore be taken up again and completed.

*Microsublimation.*—Extended studies of microsublimation in its relation to food and drug analysis were undertaken. A critical study of the types of apparatus used in this work has resulted in the development of improved apparatus and methods. A general paper discussing the value of microsublimation in food and drug analysis is under consideration. The ready sublimation has been effected of santonin from worm seed, oxalic acid from calcium oxalate in beans, of quercetin, obtained from the cotton plant, of sapogenin, obtained from Agave saponin, and of a number of other substances. It is contemplated to prepare brief notes recording the observations made.

Studies of other Vegetable Products.---1. Tepary Beans.--A brief study of Teparv beans (Phaseolus acutifolius v. latifolius), a newly discovered food product of the Indians, has also been made and a note discussing the chief characteristics is in preparation for publication.(16) 2. Coffee.--A considerable number of samples of coffee, including damaged coffee, have been examined and data collected indicating the relation of grade to objectionable material present. Microscopical changes have been observed, which, together with a specific stain (17) for the presence of mold, have proved of value in the judgment of the quality of import and interstate samples. The data obtained have helped to increase our knowledge of the nature and extent of changes which take place through maturing and through spoiling of coffee. The work will be continued. 3. Peat.—The nature of nitrogen in peat and the possible value of peat in feed products is another problem, to which some attention is being given. Experiments were undertaken to locate microchemically chitin, possibly responsible for some of the nitrogen. Considerable difficulty was experienced in breaking up and identifying the highly carbonized peat particles. Potassium hydroxide has thus far been found to be the most useful agent in breaking up peat without dissolving chitin, which is converted to chitosan and can as such be specifically stained.(17) While further experiments have to be made, the results thus far obtained are promising. Chitin likely is only accidentally present in peat, due to numerous dead bodies of insects buried in peat and containing chitin.

Isolation and Study of the Composition of the Cotton Plant, Gossypium herbaceum, and other Gossypium Species.—A volatile oil, decidedly attractive to boll weevils, has, with the assistance of Drs. Johns and Chernoff, and others, been isolated from different parts of the cotton plant. The oil is found in lysigenous glands which are located near the surface in nearly all parts of the cotton plant. Besides the oil, these glands contain flavone glucosides, if exposed to light, and gossypol, if unexposed to light.(19) Two papers, discussing the findings in detail, have been published in the Journal of Agricultural Research.(18,20).

Differentiation of Plant Species.—The value of pollen grains for the differentiation of species is under investigation. Numerous other data on the morphological, anatomical, chemical and physiological differentiation of plant species, genera, etc., have been or are being collected. These data have been very valuable in deciding questions of true relationship of plants and plant products. The knowledge of the proper classification of a vegetable product or plant, we feel, often permits a preliminary judgment as to their possible utilization for medicinal, food or technical purposes.

## COÖPERATION.

The laboratory has cooperated with the Scientific Division of the Shipping Board in formulating a revised system of classification of imports and exports of crude drugs and chemical products. Data on various products have been furnished the Price Section of the War Trade Board. Detailed and constructive criticism has been made of the crude drug portion of the import tabulation of chemical products, which was drawn up under the auspices of the Department of Commerce. Information on several drug products has been furnished the Tariff Commission. Some work of a confidential character has been carried out for the Aircraft Production Board. The laboratory has cooperated with different laboratories of the Bureau of Plant Industry, e. g., in the growing of a number of plants, such as Spanish digitalis, mustards, beans, etc.; with the Bureau of Entomology in the fight of the cotton boll weevil, and is assisting the Smithsonian Institution in the identification of certain supplies, especially Cinchona barks, secured by Dr. Rose in his exploration of Ecuador. The laboratory has further assisted with advice other institutions, such as the Bureau of Science, Philippines, the Department of Internal Revenue in Canada, or other state institutions and private parties in this country. Suggestions regarding the further improvement of the Pharmacopoeia have been submitted to the Pharmcopoeial Committee and it is hoped to continue not only this coöperation but to extend it also to the Committee concerned with the revision of the National Formulary.

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### ACID-INSOLUBLE ASH STANDARDS FOR CRUDE DRUGS.\*

BY CLARE OLIN EWING AND ARNO VIEHOEVER.

Having had occasion to review analyses of a considerable number of domestic and imported crude drugs with regard to their content of ash and acid-insoluble ash, the attention of the writers was called to a number of instances where a striking discrepancy occurred between the general run of analyses and the U. S. Pharmacopoeia and National Formulary Standards. In some instances it appeared that the present standards were somewhat rigid, whereas in others the ash standard was placed so high as to excite suspicion that a sample of the product in question yielding such an ash would be extremely dirty. It appeared, furthermore, that determinations of the ash insoluble in 10 percent hydrochloric acid would often disclose the true condition of the material, as regards cleanliness, and that an ex-

<sup>\*</sup> The changes of existing standards and the establishment or adoption of new limits as standards, suggested in this manuscript, should be considered as an expression of our personal opinion and not as an official announcement of the Bureau of Chemistry.