

Reports of A. Ph. A. Committees

THE PROGRESS OF PHARMACY.

Abstracts from the Report on the Progress of Pharmacy for the Year 1911, by C. Lewis Diehl, Reporter.

(Second Installment.)

Acetic Ether: Rational Method of Preparation.—According to A. Kurtenaker and H. Habermann acetic ether is most rationally prepared from molecular proportions of alcohol and acetic acid, using nickel sulphate as a dehydrating medium. While an excess of alcohol causes the esterification of a larger percentage of the acetic acid, the resulting acetic ether is contaminated with considerable quantities of alcohol which can be removed only by a tedious process. They recommend that the reacting mixture be heated in a water bath, under a reflux condenser, and as soon as the thermometer indicates a boiling point of about 73°, to reverse the condenser and collect the distillate so long as the boiling point does not rise materially above 73°. When this occurs, the mixture is again heated under the reflux condenser until the contents of the still again boil at 73°, when the distillate is collected as before—this alternate systematic treatment being repeated until no further distillate passes over at the temperature of 73°. This method secures the largest yield of pure acetic ether.—*Jour. f. prakt. Chem.*, 1911, No. 12.

Camphor: Tests of Identity and Purity.—W. Lenz has made some comprehensive studies regarding the tests of identity and purity of camphor. He finds that the determination of the melting point affords an excellent criterion and aid to establish the purity of a sample. The optical rotation, however, gives no reliable data for the valuation of crude camphor, the impurities of which show a stronger rotatory power than pure camphor; it is of value, however, to determine whether or not the sample consists of natural d-camphor. While the determination of the residue of evaporation is a valu-

able aid to establish the purity of camphor, this is too tedious and consequently impracticable. The conversion into oxim has been improved by the author so that a yield of about 93% instead of from 75 to 85% is obtainable; but a valuation of the sample is impracticable because of the disparity between the theoretical and actual yield of oxim is still too great. The vanillin-hydrochloric reaction is of service only for the identification of natural camphor; moreover, the red color of this reaction is exceeded by that produced by pure hydrochloric acid (38% HCl) on natural camphor. But the amount of substance insoluble in 10 parts of this strong hydrochloric acid affords an excellent criterion for estimating the impurities in commercial camphor, which may serve well as a basis for a reliable process. In point of fact, however, it may be said that no single reaction will serve to give a clear view of the composition of crude camphor, which becomes possible only by a combination of the various tests and reactions proposed.—*Arch. d. Pharm.*, 249 (1911), No. 4, 286-298.

Synthetic Camphor: Continued Improvement in German Manufacture.—Gehe & Co. report that while in other countries some of the manufacturers of Synthetic Camphor have relinquished their efforts to compete with the natural product, owing to the continued high price of oil of turpentine and the reduction in the price of Japanese Camphor, the restless energy of German manufacturers in improving and devising new methods has enabled them to compete successfully and to maintain the position of synthetic camphor on the market. The outlook for a continuation of the manufacture of synthetic camphor is the more encouraging, since it has been clearly proven that the artificial product possesses identically the same therapeutic properties and practically the same chemical character as the natural.—*Pharm. Ztg.*, LVI (1911), No. 32, 321.

Barium, Strontium and Calcium: Quantitative Separation and Estimation.—v. d. Horn

and v. d. Bos describe a new method for the quantitative separation of barium, strontium and calcium, which is adaptable both for their gravimetric and volumetric estimation in their admixtures. The dilute solution is acidulated with acetic acid and the barium precipitated with excess of ammonium chromate, the barium chromate being collected on a filter, washed, dried and weighed. From the filtrate the strontium chromate is precipitated quantitatively by 50% alcohol, and in the filtrate from this the calcium is quantitatively precipitated as oxalate. The details of the volumetric method, based upon the reactions described, must be consulted in the original abstract.—Pharm. Weekbl., 1911, 5, through Pharm. Zentralh., LII (1911), No. 30, 298.

Mercury: Colorimetric Estimation in Very Dilute Solutions.—H. R. Procter and R. A. Seymour-Jones recommends a colorimetric method for the estimation of mercury in very dilute solution which is based upon the observations that the metal is not precipitable as sulphide with H_2S in the presence of formic-, citric- or other organic acid, the HgS being retained in colloidal solution. The intensity of the color produced is in direct proportion to the amount of metal present, which may then be estimated by comparison with a standardized solution of the same by one of the known colorimetric methods.—Journ. Soc. Chem. Industr., through Chem. Ztg., 1911, No. 90.

Nitrates: Detection in Portable Waters.—Caron and Raquet recommend a solution of salicylic acid in concentrated sulphuric acid as a valuable reagent for nitrates in drinking waters. Ten Cc. of the water are evaporated to dryness, the residue is triturated with 1 Cc. of the reagent, and 10 Cc. of water, followed by 10 Cc. ammonia are added. In the presence of nitrates a yellow color is developed, which may be made available for their colorimetric estimation. It is essential that the reagent is freshly prepared, but this can be avoided by adding 1 Cc. of a 1% solution of sodium salicylate to the evaporating water under examination, and then adding simply concentrated sulphuric acid, followed as before mentioned with water and ammonia.—Rép. de Pharm., 1911, No. 6.

Nitrites: New Reagent for Their Detection in Potable Waters.—Dané proposes as reagent for nitrous acid in potable waters a

solution of 0.02 of synthetic indol in 150.0 of 95% alcohol. If 2 to 5 Cc. of this new reagent are added to waters containing nitrites, and the water is then acidulated with a 50% sulphuric acid, a rose-red color is developed in the course of one minute. The reaction is quite sensitive; it may be made available for colorimetric determinations of nitrites in waters, and serves also for the detection of nitrous acid in other reagents.—Bull. de la. Soc. Chim., IX (1911), 345.

Kolatein (Kolatein): A New Constituent of Kola-Nuts.—A. Goris describes a new, phenol-like constituent of fresh kola-nuts, which he obtained during the isolation of kolatin with which it occurs associated and is separated by its insolubility in ether, in which kolatin is soluble. By several recrystallizations the new body, which the author has named *Kolatein*, was obtained in a pure condition, leaving no residue on combustion. It does not liberate carbonic acid from $KHCO_3$, is precipitated from its solutions by lead acetate, gives a green color with ferric chloride and its solution becomes violet-red on addition of ammonia. *Kolatein* is soluble in hot water, more sparingly in cold water; also soluble in alcohol, acetone and wood-spirit but insoluble in ether, chloroform, petroleum ether and xylol. From its aqueous solution it crystallizes with water of crystallization, which, however, it loses rapidly on drying, and in this respect resembles phloroglucin with which it has various reactions in common. It differs, however, from phloroglucin by its bitter taste, its coloration (green) with ferric chloride, and its melting point, which is 257° - 258° . In its chemical relations it apparently belongs to the group of catechins.—Bull. des. Science. de Pharmacol, 1911, No. 3.

Opium: Culture-Experiments in Austria.—Dr. W. Mitlacher and R. Wasicky give some interesting information concerning culture experiments undertaken in Austria during recent years with the object of cheapening the method of collecting the opium from the poppy. It has been demonstrated by numerous experiments recorded during the past century that the poppy (*Papaver somniferum*, L.) cultivated throughout Central Europe, will yield by the Oriental method of collection (by incisions in the unripe capsules) opium differing very little or not at all from Smyrna opium of normal quality in its alkaloidal con-

tent. The cultivation of the poppy for the production of opium has, however, been prohibitive on account of the high cost of the labor required for collecting the poppy juice by incisions on the capsules of the growing plant. The authors therefore conceived that a method of expression might lead to the economical production of the opium of satisfactory quality; but in this they were disappointed. Opium obtained in this way was extremely deficient in alkaloid, containing only about 5% of the quantity of morphine found in opium prepared from the same crop by the Oriental method; nor was it possible to obtain more than 20% of the morphine content of the latter, by repeated extraction of the marc remaining after the expression of the juice. Nevertheless, the authors believe that by improving the method of expression, using a press of greater power, possibly in connection with an inflow of warm water, it may be practicable to prepare an opium with a profitable content of alkaloid, though admitting that at present the proposed method has no practical value.—Unt. d. Allg. Oesterr. Apoth.-Ver., 1911, No. 5.

Rhamnus Cathartica: Constituents of the Bark.—Introducing the subject of their recent researches on the constituents of the bark of *Rhamnus Cathartica*, A. Tschirch and H. Bromberger mention that more than 60 years ago Max Boriswanger had determined the presence in this bark of the following constituents: An oil (colored green by chlorophyll) rhamnoxanthin, amorphous resin; tannin, crystallizable bitter principle, and sugar; but none of these bodies were definitely characterized. Since then (1898), Tschirch announced the presence of "Oxymethylanthraquinone" in this bark (compare also *Rhamnus Catharticus*," Proceedings, 1901, 741). By their recent researches Tschirch and Bromberger have now isolated and definitely describe the following additional constituents: "*Rhamnosterin*," a nearly colorless body belonging to the phytosterins; "*Rhamnofluorin*," an ash-gray body, crystallizing in flat plates, soluble in ammonia and alcohol with green-yellow fluorescence; "*Emodin*" (=Frangula-Emodin); "*Chrysophanol*" (=pure Chrysophanic acid); "*S-Glucose*"; and "*Tannic Acid*." Arch. d. Pharm., 249 (1911), No. 3, 218-223.

Gentian Root: Changes During the Vegetation Period.—M. Bridel makes some inter-

esting observations concerning the changes in chemical constituents of Gentian Root during the vegetation period of one year. He finds that considerable variations occur during the period, particularly in the direction of the carbohydrate hydrolysable by inversion. In the case of gentiopicrin these variations are not appreciable, the roots containing constantly at least 2% of this glucoside. The quantity of carbohydrates hydrolysable by inversion, however, which at the beginning of the vegetation period amounts to 1.2% was found at the end to be 7.8%. Gentianose is always present in the amount of $\frac{3}{5}\%$, except during the months of May and June, at which period the root contains gentiobiose, and that the content of gentianose is greatest during the months of August and September. The greatest variations, in fact, is shown in the saccharose content, which accumulates towards the end of the vegetation period and disappears completely at the beginning of the next, as does also a large portion of the gentianose—Journ. de Pharm. et Chim., 1911, No. 6.

Tincture of Gentian: Improved Preparation.—In the course of his studies on the constituents of Gentian Root and the changes occurring during the process of drying and preservation (see *Gentian Roots*, above), Bridel has determined that the active constituents of the fresh roots may be preserved without appreciable loss if a rational process of drying is observed. In a further communication the author now points out that the ferment existing in fresh Gentian Root, being practically unchanged in the properly dried drug, it is liable to exert unfavorable activity on the gentiopicrin if the gentian is prepared in the ordinary way, and that this ferment should therefore be destroyed by preparing the tincture with hot alcohol. A properly prepared tincture of gentian should contain about 1% of gentiopicrin.—Pharm. Ztg., LVI (1911), No. 54, 544.

Cherry-Laurel Water: Preservation, Clarification, etc.—A. Astom points out that cherry-laurel water can be effectually preserved only by keeping it in completely filled, well-closed bottles, protected from light. The loss in hydrocyanic acid in open bottles, particularly when they are not filled, is very considerable, and this loss is greatly increased by the influence of light. The author advises the use of small brown-glass bottles for

storing this water and also for dispensing it. Moreover it must be tested from time to time regarding its strength, and should be renewed more frequently than is at present required by the French Pharmacopœia, which directs the renewal of distilled waters annually. Regarding the clarification of cherry-laurel water, the author cautions against the use of animal charcoal, which is capable of absorbing and holding considerable quantities of HCN. This absorption varies with different kinds of charcoal and the quantity, but time of exposure or the temperature appears to be of comparatively small influence.—*Jour. de. Pharm. et Chim.*, Vol. IV (1911), No. 1.

Extracts of Belladonna and Hyoscyamus: New Method of Alkaloidal Assay.—A new and simple method for the estimation of alkaloid in the extracts of belladonna and hyoscyamus has been adopted in the laboratory of the Association "Pharmakon" of St. Petersburg, which may be briefly outlined as follows: The extract (using twice as much extr. hyoscyam as belladon.) is liquified with water and, after addition of ammonia, vigorously shaken with a measured quantity of ether. After subsidence, an aliquot part of the ethereal extraction is evaporated, the residue is dissolved in alcohol, the solution diluted with water, and a specified quantity of 1/10 N-hydrochloric acid added, the whole being adjusted with water to a specified volume. An aliquot quantity of liquid is then filtered off, a measured quantity of ether is added, and the unconsumed hydrochloric acid is then ascertained by titration with 1/10 N-potassium hydroxide, using iodeosin as indicator, to a faint rose color. The reaction is quite sharp, and the calculation is made in the well-known manner.—*Farmaz. Journ. russ.*, 1911, 138.

Syrup of Raspberry: Detection of Foreign Coloring Matter.—Schwickard points out that the G. P. method of testing for foreign coloring matters in syrup of raspberry, which depends on their solubility in amyl alcohol, is liable to be misleading because the latter acquires a strong rose color with all natural raspberry juices. He therefore recommends the destruction of the natural color by treatment with sodium hydroxide solution before applying the amylalcohol test.—*Pharm. Ztg.*, LVI (1911), No. 57, 578.

Ferrated Cod Liver Oil: Preparation with

Ferric Benzoate.—Const. Kollo points out that the solubility of ferric benzoate in cod liver oil depends on the method of its preparation and the care with which it has been preserved, and therefore considers it best that pharmacists prepare this salt themselves to assure its proper quality, for which purpose he recommends the following formula: 6 Gm. of pulverized benzoic acid are suspended in 120 Gm. of distilled water and accurately neutralized with 10% ammonia, of which about 8.5 Gm. are required. The filtered solution is transferred into a wide and deep porcelain basin, and a solution of 9.5 Gm. of solution of ferric chloride of sp. gr. 1.280-1.282, previously diluted with 250 Gm. of distilled water and neutralized with ammonia as accurately as possible, is added with assiduous stirring. The voluminous precipitate is allowed to subside, washed until the washings no longer give a chlorine reaction, drained, and rapidly dried.—*Pharm. Post*, 1911, No. 51.

Ung. Hydrarg. Oxyd. Flav: Preparation.—M. J. Romeyer states that the following method yields a yellow oxide of mercury ointment satisfactorily conforming to all requirements: Using a porcelain mortar and porcelain or glass pestle, all these carefully cleansed with hydrochloric acid and distilled water and well dried, 5.0 Hydrarg. Oxydat. flav. and 15.0 Lanolin Anhydric. are intimately triturated with the aid of the heat of a water-bath. The reddening of the mercuric oxide under the influence of the heating disappears completely on cooling. This mixture is finally incorporated with 80.0 Vaselin. alb., 10.0 or more of which may be replaced in winter by Ol. Vaselin. pur. The product possesses permanent stability and is free from all side-effects.—*L. Union Pharm.*, 1911, No. 50.

Propolis: Production, Character, Composition, Etc.—M. Kistenmacher contributes an interesting and comprehensive study of propolis (bee-bread) which, almost completely forgotten as a remedy, has in recent years again attracted attention. He describes its significance in the hive, the method of its production by the bees from the oil or balsam on the surface of pollen grains, its influence on the construction of the honey-comb, its properties, composition, etc. In the fresh condition, propolis is a very soft mass, possessing strong adhesive power, a strongly aromatic odor, a bitter taste, and varies in

color from greenish-yellow to liver-brown. The older bee-bread, containing waxy and refuse matters, etc., usually has a darker color and is less adhesive. The propolis or bee-bread of commerce contains comparatively only a small percentage of solid matter, its composition depending upon the work of the bees themselves as well as the method of the collection by the bee-keeper. It is mainly composed of an oil or balsam—the so-called propolis balsam—which in turn is composed of cinnamic alcohol, cinnamic acid, tannins and resins.—Ber. d. D. Pharm. Ges., 1911, No. 1.

Yoghurt: Preparation.—Dr. H. Kühl contributes a lengthy paper describing the properties and preparation of the Bulgarian milk-food known as "Yoghurt" (also "jaurt") which is receiving considerable attention in recent years and was described in the "Report" of last year (see Proc. 1910, 390). The author does not add much to the description there given, but gives some practical details respecting its preparation which may be of supplementary interest. He says that of the three Bacilli that are concerned in the production of yoghurt from milk, the one of most importance is *Bacillus Bulgaricus*, since

it is principally concerned in the peculiar acidification that characterizes this preparation, the other two (*Bacterium lactic acidii* Güntheri and a lactic acid *Streptococcus*) serving mainly the purpose of modifying the taste and preparing the milk for acidification. To prepare the thin-liquid yoghurt, which is preferred to the thick yoghurt used in the Balkan states, good, fresh milk is subjected to brief boiling and then allowed to cool to 45°C., whereupon it is at once inoculated with some old yoghurt or, if this is not available, with a yoghurt culture, which must be well mixed with the milk. It is then allowed to stand at a temperature of about 40°C., observing that the temperature does not fall below 35°C., at which temperature the growth of the *Bacillus Bulgaricus* ceases.

When the milk shows signs of thickening—usually after three to five hours—the vessel is transferred to a cool place, and the yoghurt is then ready for use, retaining its good quality and taste several days. To assure its proper quality an experiment is made with a fresh portion of milk, inoculated with some of the yoghurt just prepared.—Pharm. Ztg. LVI (1911), No. 45, 454; from Südd. Apoth. Ztg., 1911, No. 43.

REPORT OF THE COMMITTEE ON UNOFFICIAL STANDARDS.

The following portion of the report of the Committee on Unofficial Standards relates to certain crude drugs and chemicals suggested for inclusion in the next revision of the National Formulary, and by order of the Council is published in the JOURNAL in order to afford opportunity for discussion before the standards proposed are finally adopted.

Manufacturers, importers, analysts, and others interested in any of the proposed standards, are requested to send their criticisms and comments to the chairman of the committee, Geo. M. Beringer, 501 Federal St., Camden, N. J.

APPROVED MONOGRAPHS SUBMITTED AS STANDARDS FOR UNOFFICIAL DRUGS AND CHEMICAL PRODUCTS.

(Continued from January issue—page 73.)

CANELLA ALBA.

CANELLA.

The dried bark of *Canella Winterana* (L) Gaertn. (Fam. *Canellaceae*).

In quills usually from 1 to 3 dm. long and 1 to 4 cm. thick, occasionally 2 or 3 times as large or in irregular fragments of such quills, the bark from 1.5 to 4 or 5 mm. thick, the

outer periderm mostly removed; outer surface light brownish-yellow or pale orange-brown, more or less scaly, with few very shallow fissures, often more or less reticulate with slight ridges; inner surface paler, smoothish, but showing coarse, longitudinal striae; fracture short and sharp, pale yellow, with an irregular slightly darker band just