Reports of A. Ph. A. Committees

THE PROGRESS OF PHARMACY.

Submitting herewith the *first installment* of Abstracts from the Report on the Progress of Pharmacy during the year 1912, it becomes necessary to explain that all references to the reports prior to the report 1911, which is now in the printer's hands, will, as heretofore, be indicated by the word "Proceedings" (1910, 1909, etc), while the references to the reports published since 1910 will be indicated by the word "Report" (1911, 1912, etc.) Similarly, references to the Journal of the Association will be indicated by the word "Journal" (Jan., Feb., March, 1912, etc.) References to the Yearbook of the Association, however, if not part of the "Report," will be indicated by the abbreviated title "Yearbook A. Ph. A."

C. LEWIS DIEHL, *Reporter.*

Ambrette Seed Oils Properties and Constants.—It is well known that the normal distillate of ambrette seed is of a wax-like consistency, due to the large proportion of highly molecular fatty acids which it contains (principally palmitic acid), and that the liquid oil, from which all odorless admixtures is removed, is obtainable only by special treatment.

Schimmel and Co. have determined the constants in both of these:

Normal, Solid Distillate: Sp. gr. at 40°, 0.891 to 0.892; acid val., 75 to 123; ester val., 66 to 113; solid pt., 38° to 39°; insoluble in 10 vol. of 90% alcohol.

Liquid Oil: Sp. gr. at 15°, 0.9088 to 0.9123; opt. rot. 0°14′ to 1°19′; refr. index., 1.47421 to 1.47646; acid val., 0 to 2.4; ester val., 167.7 to 180.5; soluble in 3 to 6 vols. and more of 80% alcohol.—Schimmel's Rep., April, 1912, 25.

Ananas Sativa: *Analysis of the Fruit and Plant.*—E. V. Flack, Government Analyst, has subjected pineapples and the plant grown in the Bathurst District of the Cape Colony, to proximate analysis, with the following results:

	Fresh Fruit	*Fresh Plant*
Moisture	83.86%	81.45%
Crude Fat	1.11%	0.47%
Proteins	0.49%	0.75%
Crude Fibre	0.33%	3.25%
Nitrogen-free Extract	13.51%	12.02%
Ash	0.70%	2.06%
Silica	0.069%	1.12%
Lime	0.047%	0.121%
Potash	0.358%	0.356%
Phosphoric Oxide	0.024%	0.029%

The pines were grown in a sandy loam overlying gravel.—Chem. News, March 1, 1912, 99.

Benzaldehyde: *Detection of Chlorine.*—Dr. G. Heyl points out some of the defects of the G. P. V. process for the detection of chlorine in benzaldehyde, which are not removed completely even by the various modifications that have been suggested by Herzog and others. He therefore suggests the so-called "lime method" for the detection of the halogen, which has proven reliable when carried out as follows: About 1 or 2 gm. of calcium hydroxide (which, of course, must be free from Cl, and is so obtainable) is placed into a porcelain crucible, 10 to 15 drops of the benzaldehyde are added, and thoroughly incorporated with the hydroxide by means of a glass rod. The mixture is covered with a thin layer of calcium hydroxide, and is then carefully heated in the open flame, finally to redness. After cooling, the contents of the crucible are transferred to a beaker, 5-6 cc. of water carefully added, followed by nitric acid in faint excess, and the solution is filtered through chlorine-free filter paper (or glass wool). In the presence of chlorine, turbidity, greater or less according to the quantity, is produced on the addition of silver nitrate solution. By this method the presence of chlorine is sharply determined in a mixture of 1 drop of benzol nonchloride with 50 gm. of pure benzaldehyde.—Apoth. Ztg. XXVII (1912, No. 6, 49-50.

Benzaldehyde: A Convenient Method of Detecting Chlorine.—Referring to the above method proposed by Dr. Heyl, which doubtless yields reliable results, Prof. E. Rupp recommends the following simple method, which depends upon the fact that substances containing chlorine, when burned upon a surface of cupric oxide, produce cupric chloride, the smallest traces of which imparts a green color to a non-luminous flame. To carry out the reaction a section of copper wire or, better, a strip of copper netting about 0.5 cm. wide (with 1 mm. meshes), is rolled closely-spirally at one end so as to form a roll about the thickness of a pea. This is drawn several times through a non-luminous gas-, benzin- or alcohol flame, so as to form a surface of cupric oxide, and until all yellow or green color (due to NaCl) disappears. The reagent thus produced, after cooling somewhat, is simply dipped into the flame and, after allowing the benzaldehyde to burn up completely out of the flame, it is introduced into the non-luminous part of the same. If this now shows green luminosity, chlorine is present in the sample, the duration and intensity depending proportionally to the amount of chlorine present and the quantity of benzaldehyde adhering to the spiral, which was experimentally found to be about 0.3 gm. in a wire-net spiral, or 0.1 gm. in a wire spiral of the dimensions indicated. —Apoth. Ztg. XXVII (1912), No. 10, 92.

Copaiba: Estimation of B-Caryophyllene as a Test for Adulteration.—By acting on B-Caryophyllene with nitrogen tetroxide (NO_2), a crystalline nitro-derivative of caryophyllene, $C_{12}H_{19}N_3O_6$, melting at 159.5°-160°, is obtained. This compound is found very useful for the detection and estimation of B-Caryophyllene in volatile oils, by Deussen and Eger, who have applied it to the examination of the following oils:

(1) Caryophyllene from clove oil, (2) Para copaiba oil, (3) Maracaibo oil, and (4) mixtures of Para copaiba oil with gurjun oil and African copaiba oil. Three grammes of the oil is dissolved in 25 cc. of absolute ether and treated with nitrogen oxide. As soon as the separated nitro-compound begins to agglomerate at the bottom of the vessel, the reaction is stopped, and the precipitate filtered off, washed with ether, dried on a porous tile, and weighed. The direction in which this reaction may eventually be of considerable value is shown by the following figures, giving the amount of crystalline nitro-compound obtained:

Oil	*Nitro-Compound*
1. Caryophyllene (from stem oil)	yielded 50-52%
2. Caryophyllene (from bud oil)	yielded 50%
3. Para copaiba oil (rotation—11.75°)	yielded 9.5-10%
4. Para copaiba oil (rotation—14.5°)	yielded 15%
5. Para copaiba oil (rotation—10.25°)	yielded 15%
6. Para copaiba oil (rotation—19.40°)	yielded 15-16%
7. Maracaibo copaiba oil (rotation—3.9°)	yielded 5-6%
8. Maracaibo copaiba oil (rotation—10.20°)	yielded 3%
9. Maturin copaiba oil (rotation—10.30°)	yielded 8-9%
10. No. 6 with 10% gurjun.	yielded 13.3-14.3%
11. No. 6 with 20% gurjun.	yielded 11.7-11.7%
12. No. 6 with 30% gurjun.	yielded 10.7-11.7%
13. No. 6 with 50% gurjun.	yielded 7.7- 8.7%

The authors recommend Turner's reaction for the detection of gurjun balsam in copaiba. This consists in dissolving 3 drops of the sample in 3 cc. of acetic acid with 2 drops of a freshly prepared 10 per cent. solution of sodium nitrite, then pouring the liquid onto a layer of concentrated sulphuric acid. Within half an hour a deep violet color develops in the acetic-acid solution if gurjun balsam be present.

The authors also recommend and describe a reliable method for the detection of African copabia, which is based on the difference in the melting points of the dihydrochloride of B-caryophyllene (60°-70°) and of the dihydrochloride of cardiene (117°-118°), produced by the action of gaseous HCl on the solution of the oil in absolute ether. In pure Para copaiba oil, the first mentioned is principally present, whereas in African oil cardiene dihydrochloride largely predominates. Chem. and Drug, May 25, 1912, 779; from Chem. Ztg., 1912, 561.

Kava Resin: Estimation in Admixtures with Sandal Oil.—The resin of kava roots has during recent years been frequently employed as an antigonnorrhoicum in combination with sandalwood oil, such combinations being usually exploited as trade-named specialties. Having frequent occasion to determine the resin content in such mixtures, Dr. Aufrecht has experimented with the object of finding a reliable method for its

estimation, the only published method being one given in the 1911 report of J. D. Riedel, by an unnamed author, which is based upon the sparing solubility of the resin in petroleum ether. Preliminarily, Dr. Aufrecht prepared an alcoholic extract, and from this by extraction with ether the crude resin, amounting to 5.70 per cent. of the kava root employed when competely dry. This crude resin had the following composition Soluble in petroleum ether, 5.05 per cent.; resin (insoluble in petroleum ether), 91.5 per cent; extractive substances, 3.39 per cent.; ash, 0.06 per cent.; volatile oil, traces. After trying the method proposed in Riedel's Report, and finding it unreliable, the author eventually devised a method which he recommends as reliable. This consists in saponifying the mixture of kava-resin and sandal oil with alcoholic potash, heating on a water bath to remove the alcohol completely, dissolving the saponified mixture in boiling water, transferring the solution and rinsings into a flask, acidifying it with dilute H_2SO_4, and distilling until the distillate passes perfectly clear. The residual acid-resins in the flask, representing both the free resin and the esterified resin, are then collected on a tarred filter, washed, and dried to constant weight. In the experiments recorded, the method is found to be correct within 1.51 per cent., as an average of three determinations.—Pharm. Ztg., LVII (1912), No. 10, 92-93.

Methyl Alcohol: Detection in Alcoholic Preparations.—A. Hellriegel, after discussing various methods for the detection of methyl alcohol in ethyl alcohol and its preparations, recommends the following simple method as being particularly suitable for the use of pharmacists. The preparation is subjected to distillation and the distillate is fractionated, the portion distilling at 64° to 67° containing the methyl alcohol. This fraction is then boiled for three hours, under a reflux condenser, with one-half its weight of quicklime, whereby the greater part of water that may be contained in it combines with the lime. The condenser is then reversed and the distillate collected in a dry flask, whereupon the boiling point of the fraction is determined. Pure oxalic acid, dried at 100°, is now dissolved in the distillate, and the solution is boiled about one hour, when upon cooling, oxalicaciddimethylester crystallizes out. The crystals are collected on a suction filter and their melting point, which should be 54°, is determined. The corresponding diethylester being a liquid, the methyl alcohol is thus characteristically differentiated from ethylalcohol.—Pharm. Ztg. LVII (1912), No. 1, 7.

Methyl Alcohol: Detection in Ethyl Alcohol.—Referring to the above method for the detection of methyl alcohol in alcoholic preparations proposed by Hellriegel, C. F. Reichhardt suggests an equally simple, reliable method, which depends on a color reaction produced by means of oxalic acid and sodium alizarinsulphonate on the respective alcohols, provided that the distillate contains not less than 90 per cent. of ethyl alcohol. The test is carried out as follows: To 2.5 cc. of the distillate, 1 cc. of NaOH (G. P. solution) is added, followed by 3 drops of a 1 per cent. solution of sodium alizarinsulphonate, and the test-tube is rotated until a *clear, blue-violet* mixture results. Then 0.3 to 0.35 gm. of dry oxalic acid is added, and the mixture vigorously shaken several times. If alcohol is present, the color remains unchanged; but in the presence of methylalcohol a dirty-violet colored precipitate, of gelatinous consistence, forms on the walls of the test-tube, changing to a yellow color in the course of a few hours.

Another equally effective method is proposed by Dr. Aufrecht, who refers also to that proposed by Hellriegel. This method is based on the observation of A. Trillat that when methylalcohol is oxidized with potassium dischromate and sulphuric acid, methylal (CH_2 OCH_3 OCH_3) is formed, and this, when heated with dimethylaniline, yields "tetramethyldiamidodiphenylmethane," which by oxidation, even in great dilutions, develops a magnificent blue color, becoming more intense on heating, while the blue color produced with ethylalcohol under the same conditions disappears rapidly on heating.—Pharm. Ztg. LVII (1912), No. 4, 32.

New Pill-Excipient: Practically Universal Utility for Massing.—P. B. Phillips discusses the difficulties in deciding upon the proper excipient to use in making pill-masses, and gives a number of examples. He has found, however, that the nearest approach to a universal excipient is obtained according to the following formula and directions:

Gelatini	℈ ii
Glycerini	ʒ ii
Pulv. sacchari	ʒ iii
Aq. dest. ad	℥ i

Place the gelatin in a tarred evaporating dish with about ½ ounce of distilled water, and allow to stand for some minutes. Next add the glycerin, heat the mixture until the gelatin is dissolved. Then add the sugar in powder and continue the heating until the product weighs 480 grains. Transfer the contents to a covered pot, and stir until cool. As the liquid begins to set to a solid, stir briskly with a spatula in such a way as to work a certain amount of air into the product. This serves the double purpose of making the preparation whiter and softer. The author has given the name "Massol" to this new excipient, which keeps well and needs no preservative.—Chem. & Drugg. April 6, 1912, 53.

Nor-Hyoscyamine and Nor-Atropine New Solanaceous Alkaloids.—F. H. Carr and W. C. Reynolds announce the discovery of two new alkaloids obtainable from solanaceous plants, namely, nor-hyoscyamine and nor-atropine, which have hitherto eluded researchers, but the chemical identity of which is established by ample proofs of their constitution.

Nor-hyoscyamine ($C_{16}H_{21}NO_3$) differs from hyoscyamine ($C_{17}H_{23}NO_3$) only in that the methyl group, CH_3, is replaced by an atom of hydrogen. It was first isolated from *Scopolia jajonica,* but has since been obtained from *Datura Metel, Datura Meteloides,* and *Duboisia myoporoides.* It also occurs in *Datura fastuosa* and *Mandragora vernalis,* and probably in other solanaceous plants. Nor-hyoscyamine is crystalline, melts at 140° C., and forms well crystallized salts. Its specific rotation is -23°, while that of the basic ion contained in its salts is -33.8°. The ratio between these two figures is 1:1.47, which agrees with that of hyoscyamine. As regards

Nor-atropine, just as hyoscyamine is converted by the action of alkalies to its racemic modification atropine, so by the same treatment nor-hyoscyamine is converted into its racemic modification nor-atropine. Nor-atropine melts at 113° C., and forms a hydrate melting at 73. By the action of methyl iodide upon it atropine was synthetized, thus proving its relationship to the latter. Subjected to physiological tests, by Dr. Laidlaw of the Wellcome Research Laboratories, the two new alkaloids were found to have about one-eighth the mydriatic effect of the corresponding hyoscyamine and of atropine respectively. Finally, it may be metionned that the

Pseudo-hyoscyamine, isolated by E. Merck from *Duboisia myoporoides,* is considered by the authors to be nor-hyoscyamine contaminated with a little hyoscyamine.—Chem. and Drug., May 11, 1912, 700.

Oil of Aframomum Angustifolium: A New Volatile Oil.—Schimmel & Co. have distilled from the seed of a species of cardamom indigenous in German East Africa, received from Usambara, a volatile oil in a yield of 4.5%, which proved to be similar in every respect with the oil obtained from Cameroon-cardamoms, derived from *Aframomum Danielli,* K. Schumann, while the cardamoms now under consideration are identified by Schumann as a distinct species, namely, *Aframomum Angustifolium,* K. Schum. (N. O. Zingiberaceæ). The new oil was colorless; sp. gr. at 15°, 0.9017; opt. rot., -16°50′; refract. index, 1.46911; acid v., 0.4; ester val., 4.2; soluble in 6 vols. and more of 80% alcohol. Its aroma, however, cannot be compared with that of Ceylon cardamom oil, and owing to its high cineol content it reminds rather of cajaput oil. The quantity of oil at disposal was unfortunately too small to estimate its constitution with any exactitude.—Schimmel's Rep., April, 1912, 136.

Physostigmine: Derivatives.—It has been shown by Ehrenberg that when physostigmine is heated with alkalies in the absence of air, a new base is formed in addition to carbon dioxide and methylamine. This new base, designated—

Eseroline, has been the subject of investigation by Dr. A. H. Salway in order to gain further information regarding the constitution of physostigmine. Eseroline was found to be a mono-acidic tertiary base, which contains one nitrogen atom attached to a methyl group. It yields a hydrochloride, $C_{13}H_{18}ON_2$.HCl, melting at 212° C., and a picrate melting at 195° C. The oxidation products of eseroline are:

Rubreserine, $C_{13}H_{13}O_2N_2$, a fine, deep-red crystalline compound, formed when eseroline was allowed to absorb two atoms of oxygen in the presence of alkali. It is a neutral substance, which, however, yield salts with both acids and bases. The hydrochloride, aurichloride, picrate, and silver salt of rubreserine were described.

Coeruleserine—the so-called "eserine blue"

—obtained by the slow oxidation of physostigmine, was isolated for the first time in a pure condition, and designated coeruleserine. It dissolves in water, giving, even in dilute solutions, an intensely blue color Acid solutions of coeruleserine are dichroic, being blue by transmitted light and carmine-red by reflected light. Coeruleserine has the formula $C_{17}H_{28}O_2N_8$, and yields salts with two equivalents of acid. Its formation is undoubtedly due to condensation of the degradation products of the alkaloid.—Chem. and Drug., May 11, 1912, 700.

Storax: Modification of Assay Process for Cinnamic Acid Content.—Referring to a recent article in "Perfumery and Essential Oils," in which, after drawing attention to the unsatisfactory quality of storax imported into England during the last decade, a process for the determination of the cinnamic acid content is given. C. A. Hill and T. T. Cockling suggest certain modification of this process for reasons explained, and record figures for recently imported storax, showing that while genuine storax of excellent quality can still be obtained, others that come to the market are little better than rubbish. The modified process adopted by the authors is as follows:

Saponify 2.5 grams of the prepared storax by boiling with 25 cc. of seminormal alcoholic potash and 20 cc. of alcohol for one hour under a reflux condenser; evaporate the alcohol and dissolve the saponified mass in 50 cc. of water.

Shake this aqueous solution with 20 cc. of ether, allow to stand, and separate the ethereal layer; wash the latter with 5 cc. of water, mix the washings with the aqueous solution, and reject the ethereal liquid.

Acidify the aqueous solution and extract the mixed cinnamic and resin acids by shaking out with ether four times. Transfer the ethereal solutions, after washing them with water, to a 200-cc. flask, and distill off the ether. To the residue add 100 cc. of water, connect the flask to the reflux condenser, and boil vigorously for fifteen minutes; pour off the hot liquid through a filter, allow to cool to 15°, and collect the crystals of cinnamic acid on a counterpoised filter. Repeat the extraction with the filtrate at least three times, or until no more cinnamic acid is obtained. Press the filter and crystals between blotting paper, and either dry *in vacuo* over sulphuric acid and weigh, or dissolve in alcohol and titrate with decinormal sodium hydroxide. To the result obtained add 0.03 grams for solubility of cinnamic acid in water.

Two samples from recent importations by the "British Drug Houses, Ltd.," showed: Acid val., 112.2 and 113.1; ester val., 91.3 and 92.8; sapon. val., 203.6 and 205.9; and contained 5.07% and less than 5% of cinnamic acid respectively. These were probably adulterated with resin as well as impoverished (by the abstraction of the odorous constituents); while five other samples from the same source (all of them separate consignments) gave the following constants, proving to be genuine storax: Acid val., 58.3-76.4; ester val., 118.2-145.9; sapon. val., 194.6 to 204.2, respectively. The cinnamic acid content was: 30.68%, 27.51%, 22.25%, 21.6%, and 26.64%.—Chem. and Drug., March 16, 1912, 412-413.

Unguentum Adhaesivum ("Klebesalbe"): *Useful Formula.*—Dr. Dreuw highly recommends the following formula for an adhesive salve, both on account of its composition and consistency, which he has found extremely useful for the treatment of all chronic infiltrations of the skin, whether of exzematic or psoriatic nature:

℞	Acid salicyl	10.0
	Pyrogallol	20.0
	Liqui. carbon, deterg.	20.0
	Zinc. oxydat	20.0
	Sapon virid	25.0
	Adip. lan. anhydric.	25.0

M. D. S. Ung. adhesiv.

The ointment has a white-grey color, but soon acquires a black color on the surface, while the interior retains its grey color. Its adhesive qualities, however, are remarkable, and superior to that of any other ointment. Consequently it adheres persistently to the skin, a desideratum particularly in the treatment of eczemas.—Pharm. Ztg., LVII (1912), No. 3, 27; from D. Med. Wsch.

Vanadium Compounds: Review, with Particular References to Their Therapeutic Use.—The numerous chemical investigations of vanadium and its compounds that are referred to in the "Report" of 1911 are not alone interesting from the chemical and technical standpoint, but are reflected also in the domain of medicine by the pharmacological investigations that have been made in recent

years in the search for vanadium compounds suitable for therapeutic exhibition. Dr. Felix von Oefele and Dr. J. Bullinger, in view of the prospective importance of the metal, its alloys, and saline compounds, have now contributed an interesting review of our present knowledge of these compounds, with particular reflection upon those which have found therapeutic use. They speak of the occurrence of vanadium in nature, mention some of the uses of its alloys in technical medicine—such for example as the gold and platinum alloys of vanadium in dentistry—and then proceed to the description of a number of inorganic compounds of the metal which have been favorably mentioned as therapeutic medicaments; such, for example, as the several different modifications of vanadium pentoxide (V_2O_5); the different salts of orthovanadic acid (H_3VO_5), vanadium dichloride, respectively, which are characterized by great stability, and have on this account been exploited for a number of years past as specialties under specific trade names. Other compounds that are promising are the iodides and oxyiodides of vanadium, vanadium trisulphide, and vanadium selenide. Interesting compounds also, although no pharmacological experiments have yet been made with them, are the vanadium sulphvanadates, the vanadium oxysulphvanadates, and the vanadium sulphates.—Pharm. Zeutralh., LIII (1912), No. 1, 1-9.

Volatile Oils: Effect of Hydrogen Dioxide on Flavor and Taste.—The chemist of E. Sachsse & Co. reports the results of a series of experiments which, in view of the energetic oxidizing action of hydrogen dioxide, were undertaken to determine the effect of the latter on the volatile oils containing easily oxidizable constituents—such as aldehydes, alcohols, etc., which frequently compose the aromatic flavors of mouth washes containing H_2O_2. The experiments were carried out by adding to a mixture of 40 gm. Alcohol (90 vol. percent), 30 gm. Water, and 25 gm. Hydrogen Dioxde, 0.05 gm. of the Volatile oil, and allowing this mixture to stand two months. The *taste* of the mixture was then compared with that of freshly-prepared mixture of identically the same material—no attempt being made to compare the *odor* by reason of the great dilution. The results were as follows:

Unchanged: Anethol, Anise Oil, Star-anise Oil, Bornyl-Acetate, Eucalyptol, Eucalyptus glob. Oil, Geranium Oil, Pine-needle Oil, and Thymol.

Changed: Taste fainter than fresh—Carvacrol, Eugenol, Clove Oil, and Terpineol; decidedly changed—Geraniol (insipid, musty odor), Menthol, Menthyl Acetate (taste completely destroyed), Peppermint Oils of all sort, and Cinnamic Aldehyde (completely oxidized, without a trace of cinnamon odor or taste).—Pharm. Ztg., LVII (1912), No. 4, 34.

Pharmaceutical Formulas

PROPOSED FOR A. PH. A. RECIPE BOOK.

(Continued from page 638.)

The present installment consists of formulas for Lotions, which the writer has been collecting for years. A great many of these preparations are frequently ordered on prescriptions, or called for over the counter, but the books at the disposal of the average pharmacist do not give formulas for same.

Strange to say, the pharmacopoeias and formularies of the Continent list none or very few lotions under the title "Lotio," but generally classify them as "Aqua" or "Liquor," or "Mixtura," or "Solutio," or "Spiritus," etc., as can be seen in Formula No. 1 (JOURNAL A. PH A., p. 169) for Kummerfeld's Lotion, which has the title of *Aqua* Cosmetica.

Comments and criticisms are invited.

Respectfully submitted,

OTTO RAUBENHEIMER, Chairman.

<>

Abbreviations can be found in May JOURNAL, p. 504.

Formulas No. 1 to 22, see February JOURNAL, p. 169 to 173.

Formulas No. 23 to 30, see April JOURNAL, p. 366 to 368.

Formulas No. 31 to 41, see May JOURNAL, p. 505 to 506.

Formulas No. 42 to 50, see June JOURNAL, p. 637 to 638.