Contributed and Selected

NOTES ON CHEMICAL TESTS OF THE UNITED STATES PHARMACOPOEIA.

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(Continued from page 538.)

CHRYSAROBINUM.—As medicinal Chrysarobinum is not the individual chemical substance so named by its discoverer, but a mixture of a number, neither a chemical formula, exact solubility constants, nor a definite melting point can consistently be stipulated, as is done in the U. S. P. The color is usually darker than "pale" orange-yellow. Market products are not "entirely consumed" on ignition, but the ash should not exceed 0.25 per cent., the limit set by leading foreign pharmacopoeias. A limited number of specimens examined during the past 3 years, however, mostly yielded less than 0.1 per cent. In the identity test with fuming nitric acid and ammonia the color produced is violet-red rather than violet. The same applies to the lime water test. A test needed, not provided in the U. S. P. is one for the detection of chrysophanic acid. About 0.1 gm. of the sample is boiled with 20 cc. of water; the liquid, after filtration, should be neutral to litmus paper and should not be colored otherwise than pale yellow by a drop of ferric chloride test solution. This test is directed by several pharmacopoeias.

CINCHONIDINÆ SULPHAS.—As the melting point is affected by the presence of small variable quantities of other cinchona alkaloids, it has little value as a test of identity or of purity. The salt should not leave more than 0.1 per cent. of ash. The sulphuric acid test for readily carbonizable matter should be made with about 0.1 gm. of the salt and about 2 cc. of the acid. The results of the test for fluorescing alkaloids depends considerably on the the volume of solution examined; it should not exceed 100 cc., otherwise the test will be too severe.

CINCHONINÆ SULPHAS.—In the requirement regarding solubility in chloroform "80 parts" are to be regarded as volume parts, i. e., 8 cc., not gm., should apply to this salt test.

COCAINA.—The melting point of the U. S. P. is one given by some authorities as that of the chemically pure alkaloid. Some variation should be allowed for the medicinal product, probably 96° to 98°. The ash should not exceed 0.05 per cent.

COCAINÆ HYDROCHLORIDUM.—The melting point varies so much in different hands, by reason of partial decomposition, that determination of it as a test of purity, or even of identity, is apt to lead to misinterpretation. It is better omitted from the official tests, as the others are sufficient for establishing identity and purity. The statement that the salt "is not colored by cold sulphuric acid" applies only when the proportion of the former is rather small; a mixture of 0.1 gm. of it

with 1 cc. of the acid should not show other than a slight yellowish color. The wording of the permanganate test for cinnamyl-cocaine is somewhat ambiguous and has lead to differences in interpretation. If it was intended that the violet color should remain entirely unchanged in half an hour, the test is too stringent; if it was intended that the violet tint should not disappear entirely within that time-limit, then it is somewhat too lenient. Most of the products now on the market do not completely decolorize 0.1 cc. of n/10 permanganate under the conditions of test. For the sake of uniformity MacLagan's ammonia test is better made with 0.2 cc. of ammonia water, measured with a 1 cc. pipette, instead of 4 drops. The precipitate should form within 5 minutes, but with sufficiently vigorous stirring usually appears in a considerably shorter time. The supernatant liquid, however, is not "perfectly clear," but holds in suspension for some time minute crystals of cocaine, which interfere with its transparency, but do not give it a milky or opalescent appearance.

CODEINA.—Because of decomposition at temperatures below that given as the melting point, determination of this is valueless. To prepare a 1 per cent. solution of codeine for the ferricyanide test for morphine, acidulated water or alcohol should be used, as the alkaloid is not sufficiently soluble in cold water.

CODEINÆ PHOSPHAS.—This salt effloresces rapidly at ordinary temperatures, losing all except ½ molecule of its crystal-water. This makes it difficult to prepare it with fully 2 molecules of water (8.3 per cent.) Products of different makers, recently examined, contained 1½ to 1¾ molecules, with the exception of a crystalline, uneffloresced salt, which contained only ½ molecule. In view of this and in the interest of more uniform dosage, adoption by the U. S. P. of a salt containing not more than ½ molecule of water would seem advisable, as this is evidently the most stable form.

COLCHICINA.—The term "leaflets" in the description may lead to the inference that the alkaloid is crystalline, which is not the case. An unofficial crystalline colchicine, containing choloroform of crystallization, however, is on the market. "Amorphous scales" would seem to be a better descriptive term than "leaflets." Choloroform is an impurity of which no account is taken in the U. S. P., but which is stated to be present sometimes in large quantities. The following test has been found in this laboratory to be satisfactory for detection of excessive quantities: A mixture of not more than 0.01 gm. of colchicine, 2 cc. of potassium hydroxide solution (5 p. c.) and a drop of aniline, heated to about 100° in a test-tube, should not develop an odor of phenyl isocyanide. Colchicine is officially described as "having an odor suggesting damp hay," but is now readily obtainable free from odor, as it should be.

COLLODIUM.—The U. S. P. gives neither descriptions nor tests, which are important to those who do not make this solution themselves. In a layer of 30 mm. it should not appear more than slightly yellowish nor more than slightly opalescent. Exposed to the air in a thin layer, it should leave a transparent, tenaceous film. When collodion is stirred with an equal volume of water, a viscid, stringy mass separates; this mixture should be neutral to litmus paper. On the basis of a number of specimens examined it is recommended to adjust the limits of specific gravity to 0.765 to 0.775 at 25° and the pyroxylin contents to 5.0 to 5.25 per cent., by weight, which corresponds to about 4 gm. in 100 cc. of solution at

25°. The pyroxylin is best determined by the following, slightly modified method of the German Pharmacopoeia: About 5 gm. of the collodion are weighed in a shallow, stoppered, flat-bottomed weighing-jar. About an equal quantity of water is then added, drop by drop, with stirring to divide the precipitate into separate clots. The mixture is dried to a constant weigh at 100° to 110° and the residue weighed after cooling in a desiccator. This residue should burn rapidly with a yellow flame and it should not contain more than traces of substances soluble in either water, alcohol, or "absolute ether."

CREOSOTUM.—This product, because of its naturally variable composition, frequently fails to conform to some of the U. S. P. specifications, although of high grade. This applies particularly to the statements that a solution of 140 parts of water is not perfectly clear and that a solution of 120 parts of hot water becomes turbid on cooling, the latter being given as a test of distinction from, and absence of, "coal-tar creosote." A considerable number of specimens, in which no admixture with coal-tar distillates was detected by reliable tests, disolved clear in 140 parts of cold water and their solutions in 120 parts of hot water remained clear when cooled to about 25°. These statements, therefore, should be omitted at the next revision. There is an inaccuracy also in the statement, given as a means of distinction, that water-solutions of coal-tar creosote yield white precipitates with bromine water. Coal-tar distillates in which phenol and o-cresol predominate, may yield bulky, white or yellowish precipitates, but those consisting mainly of the higher boiling cresols and xylenols give compact yellow to brown precipitates. The specific gravities of creosote recorded during several years past have mostly been within narrow limits, namely 1.078 to 1.080 at 25°. The extreme variations were 1.075 to 1.087, a sample giving the latter figure containing an unusually large proportion, about 45 per cent., of guaiacol. As pointed out recently by J. W. England, the specification that "most of it" distils between 200° and 220° is too indefinite. A requirement that not less than 80 per cent. should distil within this range is quite feasible, as 85 to 95 per cent. of distillate is frequently obtained from market products within these limits. The requirements of the French Pharmacopoeia, in this respect, are probably too restricting. They are as follows: It should begin to distil a little above 200°; about one-fourth should distil between 203° and 209°; about one-half between 209° and 215°; the remainder below 225°.

CRESOL.—The composition of market products is very variable. Only a minor portion of a large number examined came within a reasonable interpretation of the U. S. P. requirements. Specific gravities ranged between 1.0255 and 1.0465 at 25°. Those having specific gravities above 1.038 contained more or less phenol and usually dissolved clear in less than 60 parts of cold water, while those free from Phenol invariably contained substances insoluble in water. Some samples gave on distillation but small fractions between 195° and 205°, instead of the required minimum of 90 per cent. Such products, of course, contain but little *m*-cresol, which is at present considered to have the highest disinfecting power, or of *p*-cresol, which is considered to be next in value. As regards the specific gravity, it may be said that this is not important as a criterion, since the composition is ascertained with greater certainty by fractional distillation and by solubility tests, but if it is to be retained, a wider range should be given, of perhaps

1.032 to 1.038, as has been proposed by H. V. Farr. The present limits exclude some products rich in the most valuable constituents.

CUPRI SULPHAS.—The limit test for iron, which is the most common impurity, is inconsistently stringent for a substance required to be only 99.5 per cent. pure. Presence of iron must also be considered in the choice of a method for the determination of copper. Volumetric determination by the well known cuprous iodide method gives statisfactory results. As the U. S. P. requires percentages to be calculated on the basis of the fully hydrated salt free from adhering moisture, it should be remembered that granular or powdered products are likely to be somewhat efflorsced, while crystals usually contain enclosed mother liquors, although superficially dry.

ELATERINUM.—It is not certain that a physiologically active substance of definite chemical composition has as yet been isolated from Elaterium, as a different chemical formula has been assigned to elaterin by each of four investigators. It is certain that the preparation sold as elaterin is far from uniform in composition, as it has been reported to vary greatly in efficiency. For these reasons the Pharmacopoeia cannot, as it now does, give consistently either a chemical formula, molecular weight, melting point, or quantitative solubility statements. An identity reaction given in recent reference books, which is rather more characteristic than those in the U. S. P., consists in dissolving about 0.01 gm. of elaterin in about 5 cc. of melted phenol, then adding a few drops of sulphuric acid. The solution at once becomes crimson and changes quickly to scarlet. Because of the scant chemical knowledge of elaterin, only physiological means of standardization can be considered reliable.

FERRI CHLORIDUM.—According to the official method of preparation 10 parts of solution of ferric chloride, U. S. P., should yield 4 parts of product, containing theoretically 25 per cent. of iron. In practice, a larger yield of a product containing correspondingly less iron is obtained, as the solution, when evaporated to the concentration directed, must absorb water from the air before it can form a firm, crystalline mass. Made on a manufacturing scale, the salt contains 20 to 22 per cent. of iron, but the upper figure is not often reached. The generally accepted formula for this salt, $Fe_2Cl_6 . 12H_2O$, corresponds to 20.7 per cent. of iron. It is therefore apparent that the present requirement of not less than 22 per cent. in the "dry" salt, which presumably means that allowance is to be made for water in excess $6H_2O$, is not tenable. It seems advisable to return to the specification of the U. S. P. of 1890 that it contain not less than 20 per cent. of iron.

FERRI ET AMMONII TARTRAS.—For the determination of iron it is directed to take the "dry" salt. It seems reasonable to interpret this as meaning "having a dry appearance," but it is also possible to interpret this as meaning a salt free from water of hydration. As most scale preparations of iron contain considerable quantities of the latter, difference in interpretation may cause marked differences in results of assays.

FERRI ET POTASSII TARTRAS.—In the first line of the last paragraph "dry" evidently means "of dry appearance." The quantity of hydrochloric acid should be increased to 5 cc., to prevent precipitation of potassium bitaratrate.

FERRI ET QUININAE CITRAS.—In the quinine determination there is no good reason for directing spontaneous evaporation of the chloroform. It may just as

well be evaporated on a water-bath, with considerable saving of time. At it is difficult to remove chloroform completely at moderate temperatures from amorphous alkaloids, the quinine, after evaporation of the chloroform, should be dissolved in alcohol or ether, the solvent evaporated, and then the residue dried to a constant weight at 100°.

FERRI ET STRYCHNINÆ CITRAS.—Spontaneous evaporation of chloroform in the assay for strychnine causes unnecessary loss of time, but when an elevated temperature is employed, it should be low enough at the end to prevent loss of strychnine through spattering.

FERRI PHOSPHAS SOLUBILIS.—The identity tests are in need of revision, as they fail to show definitely the composition of the preparation.

FERRI PYROPHOSPHAS SOLUBILIS.—The identity tests do not differentiate this from the preceding preparation, which could be substituted for it without detection by the U. S. P. tests. Molybdate solution may be used as a means of distinction, but it should be remembered that a small amount of orthophosphate may be legitimately present in the pyrophosphate, being formed during the manufacture, also that in contact with the reagent pyrophosphate soon changes to orthophosphate.

FERRI SULPHAS EXSICCATUS.—Not "completely" soluble in water in the sense of making a clear solution, but a 5 per cent solution should not be more than slightly turbid when made with oxygen-free water. The U. S. P. does not explicitly state how much ferrous sulphate the exsiccated salt should contain. Prepared by the official directions it contains theoretically not less than 99 per cent of $FeSO_4$, $1\frac{1}{2}$ H_2O, but allowance must be made for absorption of moisture during exposure to the air while the dried salt is powdered, transferred to containers, etc. A fair requirement would be that tiration with permanganate should show it to contain not less than 95 per cent of $FeSO_4$, $1\frac{1}{2}$ H_2O.

FERRUM REDUCTUM.—Much of the "iron by hydrogen" of the market has the appearance of being some other form, possibly high grade wrought iron. Such products are in rather coarse powder, but are still "very fine," as that term is defined in the U. S. P. They also have an unmistakable, though not pronounced metallic lustre, while the U. S. P. specifies a very fine, lustreless powder. They usually meet all the chemical requirements of the U. S. P. and have a tendency to test higher in metallic iron than those products which conform more closely to the physical characteristics given for "reduced iron" in the various pharmacopœias, including the U. S. P. The superficial physical differences between these doubtful products and such as are undoubtedly made by reduction with hydrogen are sufficiently slight to be overlooked by many and to cause differences of opinion as to whether or not a given specimen conform to the letter of the present U. S. P. specification. It should be noted, in this connection, that the official definition of "reduced iron" fails to specify any particular method of reduction, so that any powdered iron conforming to the description and standing the tests for purity and strength will be admissable. If it can be shown that reduction by means of hydrogen yields a product therapeutically superior to those obtained by other means, then the definition should clearly state that this method only should be used and tests should be devised to distinguish products so made from others. The present

assay method is adapted only to iron in extremely fine powder and has been found even less satisfactory than the mercuric chloride method of the U. S. P. of 1890. This latter method, however, in its present improved form, is the most reliable known at this time. In Treadwell and Hall's Quantitative Analysis it is given as follows: "About 0.5 gm. of ferrum reductum, in the form of a fine powder, is placed in a 100 cc. graduated flask, from which the air is replaced by CO_2, 3 grms. of solid mercuric chloride are added and 50 cc. of water. The contents of the flask are then heated to boiling, by means of a small flame, and the liquid boiled for a minute. The flask is then filled up to the mark with boiled water. After cooling to 15° the solution is again carefully brought to the mark, well shaken, and then allowed to stand in the stoppered flask until the precipitate has settled. The liquid is then poured through a dry filter and the filtrate caught in a flask filled with carbon dioxide. Of this filtrate 20 cc. are taken, acidified with 20 cc. of sulphuric acid (1:4), treated with 10 cc. of manganese sulphate solution (1 liter contains 67 gm. of Mn SO_4+4H_2O, 138 cc. of phosphoric acid, sp. gr. 1.7, and 130 cc. of sulphuric acid, sp. gr. 1.82) diluted to 200 cc. and titrated with tenth-normal permanganate solution." For all except extremely fine powders the boiling should be extended to at least five minutes.

GLYCERINUM.—While perfectly pure glycerin may be entirely "colorless and odorless," this characterization, in the U. S. P., of the best article furnished to the drug trade, is not strictly accurate, but the best grades appear colorless when viewed crosswise in a colorless glass tube 30 mm. in diameter. In large bulk they are more or less yellowish; a bluish or greenish tint would indicate the presence of coloring matter, added to mask a yellow tint. The German Pharmacopœia requires, as a test for substances added to improve the appearance, that a mixture of 5 cc. each of glycerin and diluted sulphuric acid (16 p. c.) should not become yellow when boiled. Even the best commercial grades have a faint, fat-like odor, noticeable in the cold only when a considerable bulk is examined. J. M. Starkie has pointed out that the U. S. P. specific gravity is too low for glycerin of 95 per cent. and should be changed to 1.249 at 25°. The official test with Fehling's solution for sugars and that with sulphuric acid for "readily carbonizable impurities have been criticized by glycerin manufacturers as apt to mislead. In the experience of this laboratory, however, these tests are satisfactory when properly made. A very slight deposit of cuprous oxide should be disregarded, as this frequently forms after some hours when no sugars are present, but is caused by traces of other reducing substances. With glycerin containing enough sugar to make adulteration worth while, a decided precipitation would take place almost at once. The official test for butyric acid, which is in reality a test for esters of volatile fat acids in general, has been found too severe. Tests of this kind are being replaced by leading pharmacopoeias and other authorities with limit tests, such as the following: A mixture of 50 gm. of glycerin, 25 cc. of n/10 potassium hydroxide, and 25 cc. of water, boiled for 5 minutes, then cooled, should not require less than 20 cc. of n/10 acid for neutralization, with phenolphthalein as indicator.

GOSSYPIUM PURIFICATUM.—The U. S. P. specifications require revision in several particulars. Those of some other pharmacopoeias are in some respects more

definite or more exacting. In this connection some of the requirements of these other authorities are of interest, as follows: It must be uniformly soft, i. e., free from lumps or hard flakes (German, Netherlands, Swiss, Japanese, Austrian). It must contain only slight traces of chlorides, sulphates, or calcium salts (Germ., Neth., Swiss, Jap., Aust., Belg.). Of an extract prepared with boiling water (1 in 10), 10 cc. are mixed, after cooling, with 3 drops of a 0.1 per cent. solution of potassium permanganate and a few drops of diluted sulphuric acid. The mixture should not be entirely decolorized in 5 minutes (Germ.). Similar test (Swiss, Jap., Aust., Belg.). The fibers should be at least 3 cm. long (Neth.); 3 cm. long in greater part (Swiss). Absence of foreign fibers by microscopical examination (Swiss). An extract prepared with boiling water (1 in 10) should not become colored in 5 minutes if mixed with potassium iodide solution and starch paste (Neth.). Limit of moisture, 5 per cent. (Neth.); 7 per cent. (Swiss). A water-extract (1 in 10) should not be opalescent, soapy, or colored (Swiss). Limit of ether-soluble matter (fat), 0.2 per cent. (Neth.); 0.6 per cent. (Swiss). If the statement of J. Thomann, that presence of more than 0.15 per cent. of fat prevents cotton from sinking readily in cold water, can be verified, a special test for a limit of ether-soluble matter will be superfluous.

GUAIACOL.—As this substance acquires a color on exposure to light and air, it cannot be expected to be "colorless" after having been kept for some time, but it should not appear more than slightly yellowish in a stratum of 2 cm. The official melting and boiling points are those given by some authorities for chemically pure guaiacol and are not applicable to the medicinal article. As the permitted variation in specific gravity implies allowance of some impurities, corresponding variations in the melting range, approximately 27° to30°, and in the boiling range, approximately 203° to 206°, should also be allowed. Market products often exceed these limits. In the application of the potassium hydroxide test a "nearly white" mass is seldom obtained. With most specimens tested it was more or less gray, varying with the intensity and duration of heat applied. It is probable that this test, if narrowly interpreted, would cause rejection of many excellent products. Samples failing to stand the official test for "oily hydrocarbons" are sometimes met with; two of recent date yielded liquid residues consisting of brown globules, which were partically insoluble in water, but readily soluble in acidulated water, therefore probably not hydrocarbons, but of a basic nature.

GUAIACOLIS CARBONAS.—The melting points given in the U. S. P. differ considerably from those given in most other pharmacopoeias, which also differ much among themselves. Statements of the melting point of the pure substance in chemical literature are also at variance with each other. As a determination of this constant constitutes a most important test for the detection of excess quantities of other creosote compounds, greater certainty on this point is desirable. Determinations made in this laboratory indicate that chemically pure guaiacol carbonate melts at about 86° to 86.5°, when heated at a rate not exceeding 0.5° per minute in a capillary tube. These results were obtained with a commercial specimen that was recrystallized from alcohol and from benzene until there was no further change in melting point. In view of these results and the quality of the available market products, it is recommended that the present U. S. P. limits,

84° to 87°, be retained. In addition to the official tests, the following, taken from other pharmacopoeias, may be aids for ascertaining the quality of this substance: A saturated solution in alcohol should be neutral to litmus paper that has been moistened with water. A solution of 0.1 gm. in 2 cc. of sulphuric acid should be colorless or not more than slightly yellow. Not more than 0.1 per cent. of residue should remain on incineration.

HEXAMETHYLENAMINA.—The U. S. P. gives no tests for impurities. The following have been adapted from the more recently published works to the purest products of the American market: It should volatilize, when heated, without leaving more than 0.05 per cent. of residue. A water-solution (1 in 50) should not become colored or turbid when mixed with an equal volume of hydrogen sulphide solution (heavy metals). Other portions of the water-solution, acidulated with nitric acid, should give no reaction with barium chloride for sulphates and not more than a slight reaction with silver nitrate for chlorides. No color or turbidity should be produced, if 100 cc. of a water-solution (1 in 20) be heated to boiling with 0.5 cc. of Nessler's reagent (ammonium salts or paraformaldehyde).

HOMATROPINÆ HYDROBROMIDUM.—Owing to partial decomposition of the salt at the melting temperature, the melting points obtained with the same sample by different analysts are likely to vary considerably. This is also indicated by variations in the figures given in the literature, namely 209° to 216°. The rigid figures of the U. S. P., 213.8°, should therefore be replaced by flexible limits, with the understanding that the melting point is not to be considered a test of purity. Attempts to separate small quantities of the free alkaloid in a form suitable for determination of its melting point, have so far been unsuccessful, either by the official directions or other means; on evaporation of the solvent a semisolid amorphous residue was always obtained, which did not solidify on standing.

HYDRARGYRI CHLORIDUM CORROSIVUM.—The official test for "many foreign salts" is undesirable as well as superfluous, as these impurities are determined more conveniently and accurately by their insolubility in alcohol and by their remaining as a residue when the salt is volatilized by heat. The salt should not contain more than 0.1 per cent. of non-volatile matter. The test for arsenic is unnecessarily elaborate and time consuming, if the uses of the salt be considered. The simpler test of the U. S. P. of 1890 answers every practical purpose, if a test is considered necessary. Of 12 other pharmacopoeias examined not one gives a test for arsenic. For an assay, precipitation and weighing of the mercury as sulphide is doubtless the most satisfactory of the methods requiring no special apparatus and is generally considered more trustworthy than the various volumetric methods so far proposed. The convenience and accuracy of electrolylic determinations of mercury is well known. However, as a routine test, a quantitative determination of mercury is hardly required, as the percentage of mercuric chloride can be indirectly determined from the quantity of alcohol-insoluble matter, which includes all of the usual impurities this salt contains.

HYDRARGYRI CHLORIDUM MITE.—Although required to be only 99.5 per cent. pure, this salt almost always contains less than 0.1 per cent. of impurities. However, the acetic acid test for ammoniated mercury is too stringent for any of the

wellknown brands of calomel in this market, but all products, to be acceptable, should stand the following: If 0.5 gm. of the salt be heated in a test tube with 10 cc. of potassium hydroxide test solution, the vapors should not be alkaline to litmus paper. The tests for "foreign salts" and "foreign metals" are advantageously replaced with volatilization of about 2 gm. of the salt in a porcelain crucible, when not more than 0.05 per cent. of residue should remain. As any specimen that stands the official tests, or nearly so, cannot well fail to contain the required minimum percentage of mercurous chloride, a quantitative determination of mercury should usually be unnecessary. The volumetric methods so far published cannot be considered as accurate as the well-known gravimetric methods, but results accurate within 0.3 to 0.5 per cent. may be obtained by Hempel's method, slightly changed as follows: 50 cc. of n/10 iodine are added to 0.8 to 1 gm. of the sample, contained in a glass-stoppered flask of 250 to 300 cc. capacity. When the salt is moistened throughout, a solution of 2 gm. of potassium iodide in a few cc. of water is added and the mixture *at once* briskly shaken in the stoppered flask until solution of the mercury compound is complete. The excess of iodine is then titrated with n/10 sodium thiosulphate, with starch solution as indicator. each cc. of n/10 iodine (O=16) consumed corresponds to 0.02361 gm. of HgCl.

HYDRARGYRI IODIDUM FLAVUM.—The official tests fail to provide for calomel as an impurity and adulterant. Excessive quantities of it are perhaps most conveniently detected by a determination of the mercury, which should range between 60.9 and 61.5 per cent. in a salt of U. S. P. standard. Hempel's method, as above described, is applicable.

HYDRARGYRI IODIDUM RUBRUM.—The required minimum of 98.5 per cent. of $Hg I_2$ is unnecessarily low, as the impurities in a carefully made salt should not exceed 0.1 or 0.2 per cent. Complete solubility in alcohol should be an additional requirement, to exclude calomel and red mercuric sulphide. With this addition, the U. S. P. tests are sufficient to establish the purity of this salt without quantitative determinations of mercury and iodine.

HYDRARGYRI OXIDUM FLAVUM.—In making the test with oxalic acid it is important that the oxide be quite free from small lumps, as these are not readily converted into the white oxalate. The test is, however, fallacious in this respect, that red mercuric oxide can be powdered so fine that its color will not only be the same as that of precipitated mercuric oxide, but that it will also be readily changed to oxalate under the conditions of the test. The mercury in mercuric oxides is determined accurately and quickly by titration in nitric acid solution with sulphocyanate, the procedure being the same as with silver determinations according to Volhard's method. Each cc. of n/10 sulphocyanate (O=16) corresponds to 0.01083 gm. of Hg O. In presence of more than traces of chlorides the method is not exact.

HYDRARGYRI OXIDUM RUBRUM.—This product should be required to contain not more than 0.1 per cent. of non-volatile matter. The official test for "absence" of nitrate is not delicate enough to detect traces, nor is it practicable to make this product free from nitrate, but it should stand the test as now given.

HYDRARGYRUM.—More stress should be laid on complete solubility in nitric

acid, as a test for tin and antimony, and on the residue after volatilization, in connection with which "no appreciable residue" should be defined. Purified mercury of the market contains 0.02 to 0.05 per cent. of non-volatile matter.

HYDRARGYRUM AMMONIATUM.—It is not "wholly" volatilized by heat, but the residue should not exceed 0.05 per cent. Solution of the compound in diluted hydrochloric acid, precipitation and weighing of the mercury as sulphide has been found a satisfactory method of assay.

HYDRARGYRUM CUM CRETA.—The test for mercurous oxide should be made with the same proportions of materials as are directed in the test for mercuric oxide. A requirement of not less than 37 and not more than 39 per cent. of mercury should probably be made and a method of assay added. Dissolving in nitric acid, evaporating to dryness on a water-bath with a decided excess of nitric acid, then taking up in acidulated water and precipitating a mercuric sulphide, has been found a satisfactory method.

(To be continued.)

THE PRESERVATIVE ACTION OF ESSENTIAL OILS.

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INTRODUCTION.

The present status of food preservatives in this country is a peculiar one. The past few years have shown that the addition of benzoate and salicylate of soda to food preparations is frowned upon, even if the chemicals themselves are not — in the opinion of some food experts — of a highly deleterious nature. Nor is the use of inorganic preservatives viewed with much favor. Copper salts, the sulphites, the fluorides, boric acid, all have had their detractors and all of them are gradually leaving the formulae of the manufacturers. Added preservatives of this type may, therefore, be considered (temporarily at least) under the ban.

And yet, foods, beverages and pharmaceutical products which act as culture media for various bacteria and which will permit the growth of mold must of necessity be preserved in order to make them articles of commerce. There is indeed one class of natural products which has for ages past been used perhaps unwittingly by house wives and manufacturers in the preservation of their food stuffs. We have reference to the spices, and especially to cinnamon, mustard and cloves, which, as a recent investigation by Hoffmann and Evans[1] has shown — are highly preservative in their action towards spore forming bacilli and the yeasts. It seems quite reasonable to assume that the preservative nature of these

[1] J. Industrial and Eng. Chemistry, Nov., 1911, 835.