both simple and easy, inasmuch as it does not require the same amount of attention that the Marsh test requires. It also lacks the danger of explosion and injury attendant on the Marsh test.

The extreme delicacy of this reaction might be considered an objection for the reduction of the silver nitrate with the gas delivered from nearly arsenic-free zinc and sulphuric acid; but this is never as pronounced as when the minutest amount of arsenic is present.

At most the reagents give a coloration at the end of the delivery tube, but never in the flask nor throughout the solution.

It seems best to run the reagents for ten or fifteen minutes before introducing the suspected substance, and noting the difference, if any, which may occur.

SUMMARY OF DRUG EXAMINATION RESULTS.*

J. ED. BREWER.

The following substances were examined during the past twelve months in the analytical department of Smith, Kline & French Co. Instances of adulterated and inferior drugs are given, as well as comparisons between drugs of medicinal and so-called garden variety:

Acacia. Three samples of acacia siftings were examined which yielded ash in excess of the U. S. P. limit of 4 percent.

Senna. Two samples of senna siftings yielded 16.22 percent and 25.25 percent of ash respectively.

The abnormal ash in this case, as well as in the acacia siftings, is quite probably due to the fact that when the drug is sifted the fine foreign material, such as sand, pebbles, etc., from the entire drug is left almost wholly in the siftings.

Some trouble was experienced in obtaining check results in determining the ash of senna siftings, due to the presence of pebbles of considerable size. This difficulty was overcome by reducing the entire sample to a No. 40 powder.

Aloin. Three samples of aloin were examined, none of which answered all of the U. S. P. requirements. Sample No. 1 contained 2.54 percent of water insoluble material, 21 percent of alcohol insoluble material, and left a residue of 1.2 percent upon ignition. Sample No. 2 contained 0.3 percent of water insoluble material, 22.9 percent of alcohol insoluble material, and left a residue of 0.56 percent upon ignition. Sample No. 3 was not soluble in 55 parts of acetone and left a residue of 0.4 percent upon ignition.

Considerable difficulty has been experienced in obtaining aloin of U. S. P. quality.

Cudbear. One sample of powdered cudbear was examined, which yielded 60.6 percent ash, 95.8 percent of which was sodium chloride.

Sarsaparilla Root. One sample of sarsaparilla root siftings yielded 44.49 percent of ash.

Spigelia. Two samples of spigelia were examined. One of the powdered

* Presented to the Pennsylvania Pharmaceutical Association, June, 1913.

drug yielded 32.65 percent of ash and 25.4 percent of hydrochloric acid insoluble ash.

The other, of the whole root, gave 41.78 percent of ash and 37.55 percent of hydrochloric acid insoluble ash. The high ash content of this drug is probably due to the earth which adheres to the roots when they are gathered.

Anise Seed. Five samples of anise seed were examined, which contained 1.22 percent, 2.13 percent, 3.93 percent, 4.09 percent and 5.87 percent, respectively, of foreign material such as stems, sticks, dirt and considerable coriander seed.

Cardamom. Four samples of powdered cardamom were examined, none of which answered the U. S. P. requirement of not more than 4% ash. Two of these samples were deficient in the volatile oil content as they did not contain quite 1% of volatile oil.

One sample was composed of the powdered whole fruit which partially accounted for its high ash and low volatile oil content.

Guaiac. One sample of guaiac contained 34.1% of alcohol insoluble material and yielded an ash of 4.8%.

Jalap. Five samples of jalap were all of U. S. P. quality. No trouble has been experienced in obtaining jalap which answers the U. S. P. requirements, but it has been a very difficult matter to obtain two samples from the same lot which will assay the same. This difficulty is apparently caused by a great variation in the resin content of individual tubers.

Belladonna Leaves. Of twelve samples of belladonna leaves examined only one contained less than the U. S. P. requirement of mydriatic alkaloids. One sample was found to contain several scopola leaves and these samples were of very poor physical appearance due to a large proportion of thick stems present.

Nux Vomica. Three samples of nux vomica were examined. They contained 0.95%, 1.25% and 1.06% of strychnine respectively.

Hyoscyamus. Seven samples of hyoscyamus leaves contained from 0.034% to 0.066% of mydriatic alkaloids. It is almost impossible to obtain hyoscyamus leaves which will answer the U. S. P. requirement of not less than 0.08% of mydriatic alkaloids.

American Cannabis. The most active of the four samples examined had only about 4-5 of the physiological activity of a standard indian cannabis.

Santonica. Three samples of santonica were examined, two of which contained only the slightest trace of santonin and the other contained 1.93%. The National Standard Dispensatory states "Santonica should contain from 2.5% to 3.5% of santonin; yet the commercial article rarely yields more than 2%, often less."

As the above results would indicate, there is a considerable amount of santonica on the market which is absolutely worthless in so much as its anthelmintic action is concerned.

In the estimation of santonin in Santonica by Thaeter's Method as given in "Archiv. der Pharmacie," Vol. 238, page 383, precautions should be taken so that resins or a mixture of resins and santonin are not weighed as pure santonin.

Manaca. Two samples were examined, the one proved to be a mixture of about equal parts of Manaca and some foreign root and the other was composed entirely of the foreign root, the identity of which we have not ascertained.

The chief points of differentiation of these two roots has been discussed by

Mr. F. A. Miller in Vol. 2, No. 5, page 594 of "The Journal of the American Pharmaceutical Association."

Parsley Seed. One sample of medicinal parsley seed was compared with four samples of garden parsley seed and they were found to be identical in microscopical structure, germinating power and apiol content.

The medicinal parsley seed yielded 21.76% of acetone and 6.7% of alcohol soluble extract. The garden parsley seed yielded 21.04% of acetone and 5.2% of alcohol soluble extract.

Coca Leaves. As usual no trouble was experienced in obtaining coca leaves of U. S. P. quality as the one sample which was examined yielded 1.18% of ether-soluble alkaloids.

ANALYTICAL DEPARTMENT OF SMITH, KLINE AND FRENCH COMPANY.

REPORT OF P. P. A. COMMITTEE ON DRUG MARKET.*

Owing to the unusually heavy demand upon the time of your Chairman and his co-workers on the Committee on Drug Market during the past winter and spring, the report which we have to offer this year has been gathered from a somewhat more limited field than usual. We trust, however, that it will be found fairly to outline the quality of drug products, as offered in the Pennsylvania market during the past year.

Not many startling cases of willful adulteration have come to our notice, but the vagaries of climate and season have as usual played their part in influencing the quality of the vegetable drugs on the market,—tending to prove that our U. S. P. standards for the vegetable drugs, must of necessity be largely arbitrary and that it is a very difficult matter for the U. S. P. Revision Committee to fix minimum standards for drugs that are reasonably high, and at the same time are such that at some time or other during the ten years, more or less, through which they are official, are not too high because of seasonal conditions.

Nevertheless, your Committee most heartily approves of the stand which the Revision Committee has taken in opposition to the proposition to withdraw all standards for the drugs themselves, applying restrictions only to the preparations of the drugs. Such a course would result, we believe, in tending to make the United States the dumping ground for the poorer quality of all foreign drugs, just as it used to be the dumping ground,—but thanks to our efficient customs officials, no longer is, for poor quality asafœtida. We believe in maintaining reasonably high standards, and if, as occasionally happens, a drug cannot be secured of U. S. P. strength, the difficulty can be overcome by having the Treasury Department temporarily suspend its rules or lower its standard until the poor season shall have passed,—as has already been satisfactorily done in the cases of Hyoscyamus and Asafœtida.

The following specific comments on drugs and chemicals are based upon records taken from the analytic laboratory files of the Smith, Kline & French Co. and the H. K. Mulford Co., from June 1, 1912, to June 1, 1913:

Acetic Acid, U. S. P. Of the seven samples examined five answered the U. S.

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* Presented to Pennsylvania Pharmaceutical Association, June, 1913.

P. requirements in every respect. The other two were satisfactory with the exception that they contained an abnormal amount of empyreumatic substances.—Reported by J. G. Roberts.

Alcohol, U. S. P. Of 75 lots examined, all tested 95% or over, and it was not necessary to reject any for any cause.—Reported by J. C. McCaffrey.

Alcohol (distilled from cane). This lot was slightly under strength and had a molasses-like odor which rendered it unfit for many pharmaceutical purposes. It did not answer the U. S. P. tests for the absence of amyl alcohol, non-volatile impurities and carbonizable or organic impurities.—Reported by J. G. Roberts.

Aloes, U. S. P. Of 15 samples examined, 10 contained less and 5 more than the 10% of moisture allowed by the U. S. P., the highest testing 13.2% and the lowest 6.9%. None of the samples gave more than 2 gm. of insoluble residue on applying the U. S. P. boiling water test, but in no case could the resulting solutions be said to be "nearly clear" as the U. S. P. directs. Neither were "nearly clear" solutions in alcohol obtained in the test for "absence of gum, dextrin and inorganic impurities," thus showing the need for an improvement in the new pharmacopœia of the wording of these requirements. Such statements as "nearly clear" are not sufficiently definite for a book of standards.—Reported by Geo. E. E'we.

Aloin, U. S. P. All of the seven samples gave residues upon ignition, ranging from 0.31% to 1.09% (U. S. P. requires no residue). All of the samples were faintly acid to litmus except one which was neutral. The lowest alcohol solubility was 98.5%. All of the samples dissolved in water to a faintly cloudy solution. All this shows that the present U. S. P. requirements for a medicinally satisfactory aloin are probably a little unnecessarily severe.—Reported by George E. E'we.

Two samples were examined and were found to be insufficiently soluble in alcohol or acetone and yielded a residue upon ignition. One sample was also not sufficiently soluble in water and gave a yellow color when shaken with petroleum ether.—Reported by J. G. Roberts.

Almond Meal from Bitter Almonds. Good examples of the substitution that is still practiced in spite of Federal, State and private supervision of food and drug products are shown by the fact that four samples of what was claimed to be Almond Meal was found to be Apricot Kernel meal.

Because of the fact that the kernels had been blanched and deprived of their outer covering before grinding we were unable to state positively by microscopic means whether these samples were genuine or not. Therefore we extracted the oil and by an examination of it we were able to determine that the samples were not Bitter Almond Meal.

An average of three samples submitted by one dealer yielded 19.9% of oil. The sample from another dealer yielded 19.4%. Wiley, in his book, "Food and Drugs," states that Almond Meal should yield about 40% of oil.

The acid number of each sample was within the limits for almond oil and the saponification number conformed to the U. S. P. standards for almond oil. But no evidence of substitutions could be established from these figures as the acid number and the saponification number of almond oil and apricot kernel oil are almost identical. The most conclusive evidence that the extracted material was

not almond oil was obtained from the iodine number which was 107 in one case and 108 in the other. This is beyond the U. S. P. limits of 95—100 for almond oil and is very close to the maximum of 108 for apricot and 109 for peach kernel oils.

In view of the fact that none of these samples responded to Bieber's test for almond oil and also because of the high iodine number and low oil content we concluded that these samples were not as represented and were probably ground from apricot kernels.—Reported by J. G. Roberts.

Alum Exsiccated, U. S. P. The moisture in 13 samples averaged 5.00% and ranged from 2.6% to 9.5%. The maximum allowed by the U. S. P. is 1%. The fact that dried alum is exceedingly hygroscopic accounts for the slight excess of moisture, but shows the necessity for being on the lookout to avoid paying for water when dry or nearly dry product is wanted. Stocks are not the only commodities that are occasionally "watered." Reported by J. C. McCaffrey.

Ammonium Iodide, U. S. P. No trouble due to decomposition was experienced with the lots examined during the past year. All of the samples examined were normal in color and remained permanent.—Reported by J. G. Roberts.

Ammonia Water, 10% U. S. P. Of 27 samples examined, 3 were below 10%, the lowest being 9.6%, 23 were above 10%, running up to 11.0%, and one was exceptionally high, running 13.4%.—Reported by Geo. E. E'we.

Ammonia Water, 26% U. S. P. Of 10 samples examined, 6 were above and 4 below standard, ranging from 25.9%-29.5%.—Reported by Geo. E. E'we.

Aniline Colors. All of the samples examined were free from arsenic by the Modified Gutzeit test, using 0.5 gm. samples; contained traces of iron but no other heavy metals by U. S. P. time limit test, using 0.5 gm. samples. One sample contained a large quantity of granular sodium chloride which may have been an adulterant, but more likely was employed by the manufacturer to render his product uniform in coloring power.

The Food and Drugs Act Regulations limit the use of aniline colors in food products to a certain list of approved colors given in Food Inspection Decision No. 76, but place no restrictions upon aniline colors used in medicines other than that they must not contain arsenic, zinc, or other harmful or poisonous constituents.—Reported by C. E. Vanderkleed.

Annatto. One sample was examined which yielded 21% of ash. This ash was principally sodium chloride which, according to some authorities, is added to intensify the color. It seems to be the general custom to do this as the samples reported by various operators all yield a high amount of ash which is practically all soluble in water.—Reported by J. G. Roberts.

Anthraquinone Green. In order to determine if this preparation contained abnormal amounts of arsenic it was subjected to Zehner's test which indicated a trace. This result was then confirmed with the Magnesium Ammonium Arsenate precipitation method given in Circular No. 102, published by the U. S. Department of Agriculture. Five parts per million of arsenic (As) was found with this method.—Reported by J. G. Roberts.

Apiol (Fluid Green). The specific gravity at 25° of three samples examined ranged from 0.986 to 1.004. All of the samples gave a turbid solution with olive oil and were miscible in all proportions with Chloroform, Alcohol and

Ether. But upon standing over night a flocculent precipitate was observed in each case.—Reported by J. G. Roberts.

Arsenic Trioxide, U. S. P. Of 8 samples examined, 2 were slightly below the required 99.8%, assaying 99.2 and 99.4% respectively.—Reported by Fritz Heidlberg.

Asafoetida, U. S. P. There does not seem to be any difficulty whatever in obtaining Gum Asafœtida that conforms to the U. S. P. requirements. The alcohol soluble portion is usually above 65% and ranged from 64.5% to 82.33%. The ash content is also well below the maximum amount permitted by the U. S. P. The highest amount of ash that we obtained on 13 samples was 11.99% and the lowest 2.91%.

The powdered asafœtida has also improved in quality. Owing to improved methods in drying it is now possible to get powdered asafœtida containing 60% to 65% of alcohol soluble matter instead of 40—50% as formerly. This powder may be a little darker in color than formerly supplied, but it is of better quality.

In spite of the general improvement in quality of Gum Asafœtida there is no doubt that good quality asafœtida has been refused admittance to the country on account of the enforcement by the Federal Government of the lead number standard. There has been considerable discussion of this subject and the concensus of opinion, both in this country and abroad, is that the government standard of not less than 200 is too drastic. A paper by Harrison and Self read before the London branch of the Pharmaceutical Society of Great Britain contains the results obtained on 37 samples of Asafœtida which the authors stated they had every reason to believe were genuine. Only four of these samples gave lead numbers above 200. Twenty-nine gave lead numbers ranging from 102—175. The remaining four gave the following numbers: 95, 59, 57 and 18. Two samples examined in the Analytical Department of Smith, Kline & French Co. gave lead numbers of 142 and 216. The source of these samples is not known, as they were obtained through a brokerage house.—Reported by J. G. Roberts.

Results on Asafœtida obtained in the H. K. Mulford Company laboratories were not so satisfactory as those reported by Mr. Roberts, but we attribute a part of the trouble to difficulty in sampling. We have recently installed a new grinding machine for gum samples made by Werner & Pfleiderer Co., Saginaw, Mich. This little machine is built on the lines of the dough-mixer and will grind a two-pound sample, organic and mineral matter alike, to a thoroughly uniform pulp in a few minutes.

Of 17 samples examined, only 8 exceeded U. S. P. requirements in alcohol-soluble matter, varying from 50 to 75.7%—while the other 9 ranged from 22.9 to 45.8%. In ash, only 7 ran under the U. S. P. limit of 15%, the other 10 running from 18.7 to 68%.—Reported by Vanderkleed & E'we.

Balsam of Fir. See paper by J. G. Roberts.

Balsam of Peru, U. S. P. Owing to the disposition in some quarters to substitute an artificial for a genuine balsam, it has become necessary to exercise the greatest precaution in purchasing and examining this product. It is claimed that the fictitious product conforms to all the U. S. P. requirements,—hence it cannot be taken for granted that because a balsam answers the U. S. P. requirements, it is a genuine article.

Two samples were examined and both gave negative results when subjected to various tests for the presence of synthetic balsams. But neither of them answer all the U. S. P. requirements. In fact we have been informed that the genuine balsam does not as a rule conform to the requirements of the U. S. P. This statement seems to be true as evidenced by the results obtained on these two lots. Both samples contained an excess of acid resins according to the U. S. P. test and one lot had a specific gravity of 1.155 at 25° C. The U. S. P. limits are from 1.140 to 1.150.

We have been informed from reliable sources that the specific gravity of a genuine Balsam of Peru usually ranges from 1.13 to 1.16; that the cinnamein content is between 50% and 60% and that it should require 2 to 3 cc. of half normal alcoholic potassium hydroxide solution for neutralization instead of not more than 2 cc. as specified by the U. S. P.—Reported by J. G. Roberts.

Balsam of Tolu, U. S. P. One lot was considered unsatisfactory because it gave indications of the presence of unknown foreign resins or exhausted balsam when subjected to a test given in the British Pharmacopœia. Sample had a dark reddish brown color.—Reported by J. G. Roberts.

Belladonna Stems. A sample assayed 0.253% mydriatic alkaloids. This sample is of academic interest only, as it could not legitimately be used for the manufacture of belladonna preparations.—Reported by W. H. Orrick.

Benzoin, U. S. P. The 39 samples examined averaged 73.5% alcohol soluble matter, ranging from 49.5% to 94.0%. The U. S. P. makes the rather indefinite requirement that Benzoin shall be "almost wholly soluble" in 5 parts of warm alcohol. (The proposed test for the new Pharmacopœia requires a 75% solubility for the Sumatra and a 90% solubility for the Siam variety, without limiting the amount of alcohol used.) The ash of all the samples was within the U. S. P. limit of 2%, except in 3 lots which ran 2.02%—2.06% and 4.68% respectively.—Reported by Geo. E. E'we.

Bone Ash. One sample sold as bone ash was really calcium phosphate, was pure white and assayed 98.8% $Ca_3(PO_4)_2$.—Reported by T. Liberati.

Blue—Soluble Laundry. About 0.5% of water insoluble material was obtained from each of three lots.—Reported by J. G. Roberts.

Brandy, U. S. P. One lot examined which contained an abnormal amount of oak tannin.—Reported by J. G. Roberts.

Boric Acid and Buchu Comp. Tablets. That medicinal tablets do not always contain what the label calls for is well illustrated by the following example. We had occasion to examine a sample of tablets labelled to contain in each tablet 2 grains each of Boric Acid and Sodium Bicarbonate, 1 grain each of Extracts of Buchu and Dog Grass, ½ grain each of Extracts of Corn Silk and Hydrangea, and 1/500 grain of Atropine Sulphate. Each tablet weighed on the average only 5.95 grains, whereas the medicinal ingredients claimed, amounted to slightly over 7 grains, exclusive of excipient. This is clearly a case of fraud. We are glad to state, however, that the tablets were not manufactured in Pennsylvania.—Reported by Geo. E. E'we.

Caramel. The 5 samples were free from carbonate, which is an abomination in preparations which are faintly acid.—Reported by Geo. E. E'we.

Catechu (Gambir), U. S. P. Two of the 6 samples examined were below

70% soluble in alcohol, assaying 39.7% and 48.6% respectively; the other four ranging between 84.6% and 88.9%. The ash of the 6 samples was within the U. S. P. limit of 5%, except in one lot which reached 6.5%.—Reported by Geo. E. E'we.

Charcoal-Animal, U. S. P. Two lots were examined; one answered the U. S. P. requirements for carbonization and ash, while the other was not completely carbonized and yielded over 4% of ash.—Reported by J. G. Roberts.

Chinese Cantharides. Two samples assayed 0.91 and 1.03% respectively of cantharidin, thus running considerably higher than the variety sold as Russian which has averaged in our laboratory during the past year 0.6% (21 samples). Powdered Chinese Cantharides differ in appearance from the Russian only in color, being brown instead of green. Aside from this, there would seem to be no reason why the Chinese variety should not be employed as its vesicating power as tried out in veterinary practice is good. The new Pharmacopœia will provide an assay process for Cantharides with a minimum standard not yet decided upon. —Reported by E'we and Vanderkleed.

Chloroform, U. S. P. Only 1 of the 12 lots was strictly U. S. P., 11 contained negligible traces of impurities decomposable by H_2SO_4, a few contained negligible traces of chlorides and chlorinated products, and one left a disagreeable odor on evaporation.—Reported by Richard Stockinger.

Two of three lots examined contained slightly abnormal amounts of decomposable impurities, otherwise they were all of U. S. P. quality.—Reported by J. G. Roberts.

Codeine Sulphate, U. S. P. Two of the samples examined were slightly effloresced, causing assays of 101.1 and 102.6% of the crystallized salt.—Reported by Geo. E. E'we.

Copaiba, U. S. P. The 10 samples examined were all within the U. S. P. limits as regards specific gravity, ranging from 0.966 to 0.987 at 25° C. (U. S. P.= 0.950—.995 at 25° C.). They were all above the U. S. P. limit of 50% resin, ranging from 54.0% to 65.9%. In the U. S. P. test for "presence of a normal proportion of acid resin," the 10 samples required from 2.62—3.20 cc. N/2 alcoholic KOH, all within the U. S. P. limits of 2.3—3.2 cc.—Reported by Geo. E. E'we.

Creosote, U. S. P. The 10 samples examined were U. S. P. except that 2 contained small quantities of "coerulignol and some other high-boiling constituents of wood tar." The specific gravity of the samples ranged between 1.078-1.081 at 25° C.; between 80.1% and 94.4% distilled between 200—220° C.; and the samples answered all other U. S. P. requirements.—Reported by Geo. E. E'we.

Cresol, U. S. P. Until recently it has been almost impossible to obtain Cresol which conformed to the specifications given in the U. S. P. It appears that the U. S. P. standard of 1.036 to 1.038 is too high, as one-half of the samples examined had a specific gravity below 1.036. Of the fifteen samples examined but seven had a gravity between the limits previously quoted. The gravity of the other samples ranged from 1.0277 to 1.0345. One had a gravity of 1.0426 but it was of exceptionally poor quality and is not to be considered.

It has also been difficult to obtain Cresol with the proper boiling point. The U. S. P. states that 90% should distil between 195° and 205° C. Seven samples

had distilling points agreeing with the U. S. P. requirements, seven others yielded 86 to 88% and the remaining one 87%.—Reported by J. G. Roberts.

Of 11 samples examined only 2 exactly met all U. S. P. requirements. Three samples only had specific gravities within the U. S. P. range of 1.036 to 1.038, 3 being lighter and 5 heavier, and only 4 distilled to the extent of 90% between 195° and 205° C., most of them distilling over in part below 195°. While the solubility requirement is of practical importance, it would seem that some definite requirement as to bactericidal power would be more important than gravity and distillation tests, inasmuch as coal-tar distillers seem to care little about collecting a distillate that exactly meets U. S. P. requirements.—Reported by Vanderkleed & E'we.

Diastase. The 2 samples examined assayed 1:300 and 1:250 respectively in starch-converting power, both being satisfactory from the standpoint of a 1:250 standard. Reported by Geo. E. E'we.

Elm Bark, U. S. P. Several samples of this material were considered unsatisfactory because they had considerable of the brownish outer bark still adhering to them. One trial sample was very inferior as it contained considerable dead wood and was partly mouldy. Reported by J. G. Roberts.

Ether, U. S. P. One sample was examined which did not conform to the requirements of the U. S. P. It had an excess of acidity, a slight foreign odor and contained an aldehyde. The U. S. P. states that a piece of moistened blue litmus paper should not acquire a red color in 10 minutes. This sample acquired a distinct red color in less than half a minute. The amount of aldehyde present was only slightly in excess of the U. S. P. limit, but a distinct test was obtained in one hour and a more decided test in 1½ hours.—Reported by J. G. Roberts.

Ethyl Chloride, U. S. P. The 21 samples examined had specific gravities ranging from 0.912 to 0.915 at 8° C. (U. S. P.=0.911—0.916), and answered all other U. S. P. requirements.—Reported by Fritz Heidlberg.

Eucalyptol, U. S. P. One sample had a specific gravity of 0.9241 at 25° C., while the U. S. P. requires not higher than 0.923 at 25° C. This sample answered all other U. S. P. requirements and was considered O. K.—Reported by Geo. E. E'we.

Ferric Chloride Solution, U. S. P. Eight of the 19 lots examined were slightly below the 10% metallic iron required by the U. S. P., ranging from 9.58% to 9.94%, the other 11 lots assayed between 10.09% and 10.64%.—Reported by Geo. E. E'we.

Ferrous Sulphate Exsiccated, U. S. P. Varies greatly in 2 $FeSO_4$ plus $3H_2O$ (which U. S. P. method of manufacture approximates). The three lots examined assayed 84.73%, 97.5% and 92.3% respectively.—Reported by Geo. E. E'we.

Formaldehyde Solution, U. S. P. Two of the 16 samples examined assayed slightly below the U. S. P. standard of 37%, testing 36.5% and 36.8%; the other 14 assayed from 37.1% to 38.7%.—Reported by Geo. E. E'we.

Gamboge, U. S. P. The 2 samples examined were strictly U. S. P.; ash 1.23 and 0.94%; alcohol insoluble matter 21.3% and 22.0%; and practically free from starch.—Reported by Geo. E. E'we.

Glycerin, U. S. P. The 10 samples examined were all above the U. S. P. specific gravity standard of 1.246 at 25° C., ranging from 1.24% to 1.251%. Most of

the samples contained negligible traces of butyric acid.—Reported by Geo. E. E'we.

As usual it has been a difficult matter to obtain a glycerin that conformed to the U. S. P. Butyric Acid test. The amount present is usually very small but it is sufficiently large to be easily detected by the U. S. P. test.—Reported by J. G. Roberts.

Guaiac, U. S. P. Four of the 15 samples examined contained less than 85% of alcohol soluble matter, assaying 63.2%, 83.5%, 70.1% and 80.4% respectively. Seven of the samples were purchased from one firm which evidently make a feature of straining their guaiac, as the seven samples all assayed above 97.3% alcohol soluble matter; the ash of these samples also ran exceptionally low, namely, from 0.07 to 1.1%.

Of the other samples, 5 contained more than 4% ash, assaying 6.8%, 8.2%, 4.9%; 4.3% and 8.4% respectively, and 3 were within the U. S. P. requirement of 4% ash, assaying 1.9%, 1.1%, 1.11% respectively.

It is practically impossible to obtain the acid number by the U. S. P. method.—Reported by Geo. E. E'we.

Honey, U. S. P. All of the samples examined were satisfactory from a chemical point of view, but several had an objectionable odor which was no doubt due to the lack of care in packing or during the time of production.

In seeking an explanation for this condition we learned that if honey is allowed to stand too long in the hive after finishing it becomes travel stained and discolored. In this manner it becomes contaminated with putrefactive substances which after a time decompose and give the honey an objectionable odor. Sometimes what is termed "brood honey" is mixed with a good quality honey. This is the portion that has been left in the hive over winter and serves as food for the bees. During this time it becomes contaminated with putrefactive substances which give the honey a rank odor and taste.—Reported by J. G. Roberts.

Hydrocyanic Acid, Diluted, U. S. P. Three of the 13 lots examined ran slightly below standard, 1.91%, 1.90%, and 1.92% respectively. The other 10 lots ranged from 2.02% to 2.40%, a rather large variation for so potent a drug.—Reported by Geo. E.E'we.

Hydrastis, U. S. P. Two lots were examined which contained only 2.45% of hydrastine. This is the first time in several years that we have had samples that contained less than the standard of 2.5% hydrastine given in the U. S. P.—Reported by J. G. Roberts.

Hydrogen Peroxide, U. S. P. Five of the 33 lots examined assayed slightly below 3% H_2O_2, the lowest assaying 2.90%; the others ranged from 3.01% to 3.30%. Two of the 33 lots required more than 2.5 cc. N/10 KOH for neutralization, 3.95 cc. and 2.60 cc. respectively.

The total solids never ran above the U. S. P. limit of 0.03 gm. per 20 cc., ranging between 0.0114 to 0.0218 gm.

Only one of many lots tested for As responded to the test, and that only contained a faint trace.

Only one of many lots tested for HFl contained this very objectionable impurity; the one lot contained considerable and was of the cheap ten cent store

variety. All of the many lots examined for heavy metals and barium were free from these.

One sample contained a small sediment of $BaSO_4$.—Reported by Carl E. Meddo.

Hypophosphorous Acids. The one sample of 30% standard examined, assayed 30.6%; 6 samples of 10% ranged between 10.7% and 11.4%; one of the four 50% samples was below standard, 47.0%; the others ranged from 50.7% to 54.7%.—Reported by Geo. E. E'we.

Iodine, U. S. P. The 13 lots examined all assayed above the U. S. P. standard of 99%; ranging from 99.1 to 99.9% absolute iodine.—Reported by Richard Stockinger.

Irish Moss, U. S. P. An examination of one lot showed that it contained 34.4% of moisture.—Reported by J. G. Rogers.

Iron Reduced, U. S. P. Of 15 samples examined, 10 assayed below 90% metallic iron and the rest above, ranging from 86.4% to 92.2%. One sample was labeled in the following interesting manner: "The quality of this product is as fine as can be practically turned out. It is not a strictly U. S. P. article because of the over-severe sulphide test." This sample produced a strong odor of H_2S immediately in the U. S. P. test for sulphide, yet many samples examined gave no blackening to lead test paper after 15 minutes in the test.—Reported by Otto Stockinger.

One lot was rejected for the reason that it did not have the proper appearance and contained an excess of sulphides.—Reported by J. G. Roberts.

Kaolin, U. S. P. None of the samples examined contained more than negligible traces of carbonates, which are a source of trouble in the manufacture of cataplasm kaolin.—Reported by Geo. E. E'we.

Kino, U. S. P. A sample assayed only 66.0% soluble in alcohol instead of being "soluble" as U. S. P. requires.—Reported by Geo. E. E'we.

Licorice Extract, Powdered. Two samples with a declaration of 30% starch assayed 39.5% and 32.3% starch respectively.—Reported by Otto Stockinger.

Lime Juice. Four samples assayed 0.0242%, 0.0269%, 0.0164% and 0.0189% respectively, of SO_2. The usual declared content of SO_2 is 0.03%.—Reported by Geo. E. E'we.

Lycopodium Substitute. Two samples of this substance were recently offered as substitutes for Lycopodium. A brief examination showed that they were merely treated starch grains colored with methyl orange.

One sample contained corn starch and the other sample potato starch. They yielded 0.77% and 2% respectively of oil. Genuine Lycopodium usually yields about 50% of oil.—Reported by J. G. Roberts.

Magnesium Sulphate, U. S. P. An excess of chlorine was found in two lots which when computed to magnesium chloride amounted to 0.42% and 1.10% respectively.—Reported by J. G. Roberts.

Milk of Magnesia. A sample assayed 20% over the strength declared on the label. This milk was recommended for the preparation of effervescent magnesium citrate solution and when so used resulted in an unpalatable preparation due to a lack of acidity.—Reported by Geo. E. E'we.

Magnesium Citrate, Effervescent. A sample so labeled contained magnesium

sulphate but no magnesium citrate, clearly a fraudulent preparation.—Reported by Geo. E. E'we.

Mercuric Oxide, Yellow, U. S. P. Three samples assayed less than the U. S. P. limit of 0.1% non-volatile matter, but two of the labels claimed only 0.005%, whereas they actually contained 0.038%. We do not understand why ridiculously exaggerated claims for purity are often made for chemical reagents of really satisfactory quality, when a conservatively accurate statement would prove more convincing.—Reported by Vanderkleed and E'we.

Myrrh, Gum. The 12 samples examined assayed on an average 36.0% alcohol soluble matter, ranging between 27.7% and 47.6%.—Reported by Geo. E. E'we.

Myrrh, Powd. The 2 samples examined varied widely in alcohol soluble matter, assaying 85.3% and 38.% respectively.—Reported by Geo. E. E'we.

Nitric Acid, U. S. P. Of the 9 lots assayed, 6 were below the U. S. P. standard of 68% HNO_3, ranging between 63.7% and 67.3%; the other 3 lots assayed 69.7%, 69.8% and 69.4% respectively.—Reported by Richard Stockinger.

Oil of Almonds, U. S. P. This oil, which was submitted as a trial sample, was abnormal in several respects. The specific gravity was a little high and the iodine number was beyond the limit specified in the U. S. P. It had an iodine number of 106, whereas the U. S. P. limits are from 95 to 100.

The mixed fatty acids melted at about 4° C. This is considerably lower than the 13° to 14° C. given by Allen in Organic Analysis, Vol. 4. The fatty acids from Peach Kernel Oil melt at 3° to 5° C.

On account of the foregoing abnormal conditions and in view of the fact that the U. S. P. Nitric Acid test indicated the presence of either peach or apricot oil, this oil was not considered genuine and was therefore rejected.—Reported by J. G. Roberts.

Oil of Aniseed, U. S. P. All of the samples were of the U. S. P. quality with the exception that one sample was optically inactive. A trace of lead was detected in three samples.—Reported by J. G. Roberts.

Oil, Castor, U. S. P. All of the samples (8) examined answered all U. S. P. requirements except the benzin solubility test, to which no sample conformed.—Reported by T. Liberati.

Oil Cloves, U. S. P. The 9 lots examined were all above the U. S. P. standard of 80% eugenol, ranging between 80.0% and 82.0%; the specific gravities were between the U. S. P. limits of 1.040-1.060 at 25° C., ranging from 1.040 to 1.056 at 25° C.; and answered all other U. S. P. requirements.—Reported by Carl E. Meddo.

Oil, Cod Liver, U. S. P. Of the 18 lots examined, 6 were slightly above the upper U. S. P. limit of 0.922 in specific gravity, ranging from 0.9221 to 0.9230; 9 were above upper limit of 150 in iodine number, ranging from 151.3 to 154.0, and 1 was below the lower iodine number limit of 140, namely, 137.3; 3 were slightly above the upper saponification number limit of 185, namely, 187, 187, and 185.4. Ten of the 18 lots examined gave orange end colors in U. S. P. test for "absence of seal oil, etc." All of the 18 lots answered all other U. S. P. requirements.

One lot had following characteristics: specific gravity 0.9200 at 25° C., iodine number 134, saponification number 186.6, orange coloration in test for seal oil,

etc.; odor very poor, answered all other U. S. P. requirements. This sample was rejected.—Reported by Geo. E. E'we.

Oil Cubebs, U. S. P. One sample marked "distilled by steam from marc of a cubebs preparation" answered all U. S. P. requirements for oil cubebs. The odor was not as pungent.—Reported by Geo. E. E'we.

Oil of Eucalyptus, U. S. P. This oil had a satisfactory appearance but it had rather an objectionable odor. Its specific gravity was slightly above the maximum limit of the U. S. P. It also was unsufficiently soluble in 70% alcohol as it required 5½ volumes to make a clear solution, whereas the U. S. P. specifies that only three volumes should be required.

In addition to the sample just discussed there was another one which answered the U. S. P. requirements with the exception that it had an optical rotation of —1° 49′ instead of the dextro-rotation of not more than plus 10°, as specified in the U. S. P.—Reported by J. G. Roberts.

Oil of Linseed, U. S. P. An examination of one sample of Linseed Oil used for core making and sold as pure oil revealed a condition of gross adulteration. No extended examination was made but mineral oil, rosin oil, and fish oil were found.—Reported by J. G. Roberts.

Oil, Olive, U. S. P. The 6 samples examined had specific gravities ranging between 0.9100 and 0.9116 (U. S. P. requires 0.910-.915); iodine numbers ranging between 81.2 and 84.2 (U. S. P. requires 80-88); 3 of the 6 had saponification numbers slightly below U. S. P. lower limit of 191, namely, 190.4, 190.4, and 190.6, the other 3 were 192, 194 and 194.5; none responded to the U. S. P. nitric acid test; and all 6 answered all other U. S. P. requirements.—Reported by T. Liberati.

Oil Orange. The optical rotation of the 8 lots examined ranged between plus 95.0° and plus 97.1° (U. S. P. equals not less than plus 95.0°). One lot marked "distilled by steam from marc of an orange peel preparation" answered all U. S. P.requirements, except of course method of manufacture, and the odor was inferior to that of an expressed oil.—Reported by Geo. E. E'we.

Oil Peppermint, U. S. P. The optical rotations of the 18 samples examined were all within 5.1° of each other, ranging from —20.7° to —26.8°, while the U. S. P. allows a range of 13°, namely, from —20° to —33°. A few of the samples contained traces of dimethyl sulphide.—Reported by Geo. E. E'we.

Oil Sandalwood, U. S. P. The optical rotations of all the samples examined during the past year ranged between the —16.0° and —20° required by the U. S. P.; the specific gravities from 0.969 to 0.971 (U. S. P. 0.965-0.980); Santalol from 92.3% to 95.2% and answered all other U. S. P. requirements.—Reported by Fritz Heidlberg.

One lot was examined which was not completely soluble in three parts of 70% alcohol as specified in the U. S. P. In connection with the performance of this test it was noted that 0.5° C. exerts an influence on the solubility; for instance, this oil was not soluble in three parts of 70% alcohol at 25° C., but a clear solution was obtained at 25.5° C.—Reported by J. G. Roberts.

Oil Turpentine, U. S. P. The specific gravity of the 11 samples examined ranged between the narrow limits of 0.859 and 0.862 (U. S. P. requires 0.860-0.870); from 84% to 95% distilled between 115°-162° C. (U. S. P. requires "the

larger part"); in the U. S. P. test for "absence of petroleum, paraffin oils or resin" from 0.0014 gm. to 0.0050 gm. residue was obtained (U. S. P. requires "a very slight residue"); and answered all other U. S. P. requirements. A certain time limit for letting the "dark mass" settle should be specified in the U. S. P. test for "absence of petroleum benzin, kerosene and similar hydrocarbons."—Reported by T. Liberati.

All of the turpentine samples examined have been of U. S. P. quality with the exception that they were not water white. They all had a light yellow color. Recent samples were almost water white.—Reported by J. G. Roberts.

Oleic Acid. A lot marked technical was examined which had a congealing point of 11.5° C. As the congealing point depends on the proportion of Palmitic and Stearic Acids present, it is quite evident that the high congealing point obtained in this instance is due to the presence of an abnormal amount of Palmitic and Stearic acids which have a higher congealing point than oleic acid.—Reported by J. G. Roberts.

Ox Gall, Powdered. None of the 7 samples examined were completely soluble in alcohol; 2, in addition were not entirely soluble in water; all gave the U. S. P. identification test, and clear solutions were not precipitated by an equal volume of alcohol. These oxgall samples seem to be simply dried and powdered oxgall, unpurified.—Reported by C. E. Meddo.

Pancreatin, U. S. P. Only one of the 7 lots examined assayed below the U. S. P. standard of 1:25, namely, 1:20, in starch converting power. One of the lots possessed a bad odor.—Reported by Geo. E. E'we.

The rejection of two lots was recommended because they had a benzin odor.—Reported by J. G. Roberts.

Papain. Of 19 samples examined 2 were up to standard (1:100), 4 were about 1:75 and 13 were about 1:50 or less in coagulated egg albumen digesting power.—Reported by L. H. Glickman.

Peroxide Bleach. A sample of so-called Peroxide Bleach submitted for examination had the following composition: Barium Sulphate 66.67%, Barium Phosphate 5.74%, Oxalic Acid 25.52%, undetermined and moisture 1.47%.

From the composition of this substance we infer that it is a by-product in the manufacture of hydrogen peroxide and that oxalic acid has been added to give it bleaching properties. This inference is strengthened by the fact that only a very small quantity of peroxide was found.

The use of the term "peroxide bleach" therefore is misbranding as it contains no peroxide other than the small quantity mentioned.—Reported by J. G. Roberts.

Pepsin Scale, U. S. P. Eighty-seven of the 95 lots examined assayed 1:3000, the other 8 lots assayed 1:2500.—Reported by L. H. Glickman.

One lot was rejected because it had a putrefactive odor.—Reported by J. G. Roberts.

Pepsin, Soluble, Powdered. Five of the 6 lots examined assayed 1:3000; the other lot assayed 1:2500. Reported by L. H. Glickman.

Pepsin, Insoluble, Powd. Nineteen of the 20 samples examined assayed 1:3000, the other one assayed 1:2500.—Reported by L. H. Glickman.

Petroleum. A sample of West Virginia Crude Petroleum was considered un-

satisfactory because it contained a large portion of lumpy material which prevented it from flowing smoothly.—Reported by J. G. Roberts.

Phenol, U. S. P. Usually there is but little trouble in obtaining carbolic acid that conforms to the requirements given in the U. S. P., but during the past six months we received one lot that had a pink color and another lot that had a low congealing point and a distinct yellow color.—Reported by J. G. Roberts.

Phosphoric Acid, U. S. P. All of the samples examined were of good quality with the exception of one trial sample which had a dark color.—Reported by J. G. Roberts.

Podophyllin. Six of 12 samples examined tested slightly below the U. S. P. standard of 99% soluble in alcohol, 98.9%, 95.2%, 98.1%, 98.9%, 97.9%, and 98.7% respectively. Three of the 12 samples assayed slightly more than the 1% ash allowed by the U. S. P., 1.2%, 1.2%, and 1.1%, respectively.—Reported by G. E. E'we.

Potassium Arsenite. The 19 lots examined ranged between 87.2% and 91.5% $KAsO_2$ plus$HAsO_2$ plus H_2O.—Reported by Carl E. Meddo.

Pumice Stone. A consignment of this material had a peculiar yellowish gray color which is quite different from the color of any sample previously examined. The National Standard Dispensatory states that pumice stone has a whitish gray and sometimes a bluish color.—Reported by J. G. Roberts.

Pyroligneous Acid. Two lots were examined and were found to contain 6.5% and 6.8% respectively of acid calculated as acetic acid.—Reported by J. G. Roberts.

Quinine, U. S. P. A sample labeled "Quinine Pure U. S. P., VIII," was anhydrous instead of containing three molecules of water.—Reported by Geo. E. E'we.

Quinine Sulphate, U. S. P. Four of the samples examined were effloresced, causing assays of 103.0-102.3-103.7 and 101.4% crystallized quinine sulphate.—Reported by Geo. E. E'we.

Rennin. A lot of rennin which assayed 1:50,000 in milk coagulating power, assayed only 1:13,000 thirteen months later. The diluent of the rennin consisted of sodium chloride and milk sugar and was free from boric acid or other preservatives. The rennin possessed an odor resembling putrid peptones when last examined.

One sample labeled "Sweet Rennin Powder—1:2500" assayed 1:2500.—Reported by Geo. E. E'we.

Sanguinarine Nitrate. Continues to assay very low; the 4 lots examined assayed 37.7%, 40.7%, 40.7% and 40.1% respectively.—Reported by W. H. Orrick.

Scammony Resin, U. S. P. The 2 samples examined assayed 65.8% and 56.7% soluble in ether (U. S. P. requires "almost completely"), but answers all other U. S. P. requirements.—Reported by Geo. E. E'we.

One lot was looked upon with suspicion because it did not give satisfactory results with the sulphuric acid identity test and also because it contained such a large amount of ether insoluble material.—Reported by J. G. Roberts.

Soap, U. S. P. The samples examined ranged from 10.5% to 27.0% moisture. —Reported by Geo. E. E'we.

Terebene, U. S. P. It seems to be impossible to obtain Terebene that conforms to the requirements given in the U. S. P. Three samples from different sources were examined. They all contained an abnormal amount of resinous substances and yielded 68 to 88% of distillate between 160° C. and 170° C. One sample had a high specific gravity and another was optically active. The U. S. P. states that it should be optically inactive and that it should be completely distilled between 160° and 170° C.—Reported by J. G. Roberts.

Yellow Wax, U. S. P. A trial sample was rejected because it gave unfavorable results with the U. S. P. paraffin and ceresin test and also because it had a low specific gravity and saponification number indicating contamination with a foreign substance.—Reported by J. G. Roberts.

The following table shows the results of 382 crude drug assays made in the Analytic Laboratory of the H. K. Mulford Company from June 1, 1912, to June 1, 1913:

Drug	No. of Assays	Lowest Assay	Highest Assay	Aver.	Standard	No. above Standard	No. below Standard
Aconite Root	10	0.272	0.800	0.433	0.5% Aconitine	2	8
Belladonna Leaves	18	0.253	0.515	0.420	0.3% Mydr. Alk	16	2
Belladonna Root	11	0.340	0.724	0.521	0.45% Mydr. Alk	8	3
Calabar Bean	7	0.095	0.196	0.146	0.15% Ether soluble alkaloids	3	4
Cannabis Indica	1	14.0	14.0	14.0	10% resin	1	0
Cantharides, Russian	21	0.292	1.130	0.604	0.6% Cantharidin	12	9
Capsicum	7	13.1	18.1	16.4	10% Oleoresin	7	0
Cinchona Red	27	5.36	10.28	7.63	5% total anhydrous alkaloids	27	0
Cinchona Yellow	1	6.34	6.34	6.34	5% total anhydrous alkaloids	1	0
Coca Leaves	4	0.353	0.928	0.713	0.5% Ether soluble alkaloids	3	1
Colchicum Corm	5	0.314	0.496	0.365	0.35% colchicine	2	3
Colchicum Seed	3	0.539	0.745	0.626	0.45% colchicine	3	0
Conium	2	0.513	0.622	0.568	0.5% Coniine	2	0
Cubebs	1	21.8	21.8	21.8	15% Oleoresin	1	0
Digitalis	20	0.213	0.450	0.316	0.25% Digitoxin	18	2
Ergot	13	0.107	0.347	0.218	0.15% Cornutine of Keller	10	3
Frangula	1	1.37	1.37	1.37	1.25% Emodin	1	0
Gelsemium	15	0.178	0.640	0.448	0.4% alkaloids	11	4
Ginger, African	17	6.85	9.92	8.42	6% Oleoresin	17	0
Ginger, Jamaica	37	3.10	5.75	4.37	4% Oleoresin	24	13
Guarana	2	4.33	4.62	4.48	3.5% alkaloids	2	0
Hyoscyamus	33	0.043	0.234	0.082	0.08% Mydr. Alk	14	19
Hydrastis	6	2.90	4.09	3.41	2.5% Hydrastine	6	0
Ipecac	15	1.73	2.63	2.13	1.75% alkaloids	14	1
Jalap	11	4.53	9.66	6.73	7% total resin	4	7
Kola Nut, Fresh	1	1.35	1.35	1.35	0.65% alkaloids	1	0
Mandrake	11	3.64	5.98	4.91	4% Resin	9	2
Nux Vomica	34	0.523	1.390	0.950	1.25% Strychnine	4	30
Opium Gum	5	10.50	12.27	11.45	9% Crystallized Morphine	5	0
Opium, Powd	13	11.95	12.75	12.26	12-12-5% Crystallized Morphine	12	1
Sanguinaria	15	2.52	6.04	4.21	2.5% alkaloids	15	0
Stramonium Leaves	14	0.170	0.618	0.297	0.25% Mydr. Alk	8	6
Veratrum	1	1.72	1.72	1.72	1% total alk	1	0
Total	382					264	118

Comparison with reports sent in previously:

Year		Total	Above	Below	Percent above
1909	Report.....	395	313	82	79.3
1910	"	340	291	49	85.6
1911	"	263	224	39	85.1
1912	"	298	235	63	78.8
1913	"	382	264	118	69.1

Last year, a lowering of the percentage of samples above standard, to 78.8% from the 85.1% of the previous year, was due principally to the poorer quality of Ergot, Ipecac, Jalap, Mandrake, Nux Vomica and Stramonium. This year's summary shows a further lowering of percentage of samples above standard down to 69.1%, due this time principally to Aconite, Physostigma, Cantharides, Colchicum Corm, Hyoscyamus, together with three of last year's offenders,—Jalap, Nux Vomica and Stramonium,—Ergot, Ipecac and Mandrake having reformed. So it goes with the vagaries of seasons.

LABORATORY NOTES.*

GEORGE E. E'WE AND CHARLES E. VANDERKLEED.

An Improvement in the Assay of Emodin-Containing Drugs.—For many years it has been customary for us to assay the emodin-containing drugs, cascara, rhubarb, senna and buckthorn, for the total amount of oxymethylanthraquinines, or emodin, yielded by the glucosides present in the drugs. While it has been known that the total cathartic action of these drugs is not due to the glucoside yielding emodin, a minimum emodin standard is undoubtedly of value in excluding inferior drugs and preparations. The method which we have used for estimating the emodin content of these drugs has already been published in these Proceedings.

Like many other assay processes, however, this one has given reliable or concordant results only when carried out under certain definite conditions. During the past year, in an effort to make these conditions uniform and invariable, we have adopted the following procedure as a substitute for the more general directions of the earlier published assay:

Sample equivalent to 0.2 gm. emodin, calculating size of sample from standard of drug and preparation. Place into 100 cc. 2 percent alcoholic KOH contained in a flask on sand (100 gms.) Boil under reflux one hour, allow to cool one-half hour, pour off liquid through cotton into cylinder. Repeat extraction three times, and evaporate in dish on water bath until nearly dry. Dissolve residue in 5 cc. water, transfer to separator, making final volume 25 cc. Add 60 cc. ether, then 10% H_2SO_4, 5 cc. at a time, until acid to litmus, then add 2 cc. more, shake for 3 minutes, allowing to separate. Draw off aqueous layer to a second separator, pour ether through cotton into a 400 cc. beaker. Repeat the extraction with 60 cc. ether three times, reject aqueous layer, evaporate the other extractions on water bath to small volume. Add 20 cc. stronger ammonia water, heat

* Presented to the Pennsylvania Pharmaceutical Association, June, 1913.