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

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on steam bath until nearly dry, add 10 cc. water, warm, add 10 cc. 10% H_2SO_4, warm 15 minutes, with almost constant stirring, cool for 15 minutes, filter on small filter into a separator. Wash beaker and filter with water until total volume is 35 cc. Place the filter paper into the beaker used in heating treatment with sulphuric acid, add 15 cc. 5% sulphuric acid, heat on steam bath with almost constant stirring for 15 minutes. Cool for 15 minutes, filter on small filter into the separator containing the first filtrate, wash the beaker and filter with water until free from acid. Total volume of both filtrates must be 70 cc. Shake out with four portions of 60 cc. ether each, evaporate in tared flask, dry at not more than 60° C. for two hours and then in desiccator to constant weight.

Boric Acid as a Preservative for Urine for Analysis.—We recently had occasion to manufacture a large quantity of Boric Acid Tablets, 2 grains, for one of the large life insurance companies, the tablets to be used by its department of medical inspection for the preservation of urine samples. We made an interesting test of the effectiveness of these tablets for the purpose for which they were intended. One tablet dissolved in four fluidounces of a urine sample preserved it for six days, whereas the control sample "spoiled" in about three days. When two tablets were used, a four-ounce sample was preserved for nine to ten days. The preserved samples remained clearer throughout, and as the presence of the boric acid does not interfere with the ordinary tests usually applied to urine samples, the value of this method is evident.

What is Terra Alba?—In the American Journal of Pharmacy for February, 1913, Professor Charles H. LaWall asks "What is Terra Alba?" Reference to older text books and dispensatories discloses the fact that in times past at least, terra alba was considered to be a silicate of aluminum. Recently an examination of fourteen samples from ten firms shows that the trade today considers it to be a non-setting form of calcium sulphate. For many pharmaceutical purposes, the present day terra alba is unsatisfactory, as it slowly reacts with carbonates in the presence of moisture, and for this reason is unsatisfactory as a diluent.

Phenol Used for Determining Bactericidal Power.—Not a few of the discrepancies that often occur in the reports of various laboratories on the phenol coefficient of disinfectants are undoubtedly due in part at least to variation in the phenol used as a standard. If the phenol used be contaminated with its higher homologues, which have greater bactericidal power, the sample is compared with too high a standard and the result of the test is too low. The assay of the supposed 5 percent solution does not provide a means for detecting the presence of higher homologues, but the U. S. P. provides a test which is serviceable for this purpose. The U. S. P. requires that phenol liquified by gentle heat, should have a congealing point not lower than 39° C., since traces of cresols, etc., tend to lower the congealing point.

A source of error may arise, however, in attributing a lower congealing point to the presence of higher phenols. We have found that a low congealing point may be due to the mere presence of moisture in amounts far short of causing liquefaction of the crystals. It is important, therefore, that the sample be first dried in a desiccator for several hours before subjecting it to the congealing-point test.

Effect of Vacuum Preservation on Precipitation.—In preserving galenical

preparations of ergot, digitalis, etc., in vacules or vacuum ampuls, it is necessary of course to place them in the vacuum containers as quickly as possible after the testing and adjustment to definite strength has been accomplished. Noting that the usual slight precipitation of inert extractive matter took place in the vacules after some days, we set about to determine whether this tendency to precipitate is increased by the vacuum treatment and if so, whether this was caused in part by a slight loss of alcohol during the evacuation of the containers. The following table shows the amount of precipitate by volume present in the same lot of preparation put up in ampuls "with" and "without vacuum" and both with and without 2 percent added alcohol.

Tests were made at intervals of one month after sealing the ampuls.

March 28	April 28	May 28
F. E. Ergot without vacuum.....	0.27%	0.34%
F. E. Ergot without vacuum, with 2% added alcohol..	0.55%	0.3%
F. E. Ergot with vacuum.....	0.28%	0.3%
F. E. Ergot with vacuum, with 2% added alcohol.....	0.3%	0.5%

It is perfectly apparent from this table that the use of vacuum in no way increases the amount of precipitation, and it furthermore shows that the addition of an extra 2 percent of alcohol in no way retards this precipitation. The ampul which contained 0.55 percent of precipitate on April 28 seems to have been a sort of exception.

The table also seems to prove that most of the precipitation occurred soon after these ampuls were filled, and that during the last month not a great deal of precipitate was added to that which originally formed.

It is needless to say that this precipitate represents only inert matter, as many tests in our laboratories have shown that these preparations retain their full activity when preserved in this manner.

Limit of Codein in Morphine.—It has been reported in several of the journals during the past year that the morphine sulphate on the market has contained considerable quantities of codein amounting in some cases to 7 percent. We have examined a considerable number of samples by the method given by Williams in the September, 1912, number of the American Journal of Pharmacy, which is practically the same method as will be incorporated in the new Pharmacopoeia. We also tested the samples by the methods of the Netherlands, Swiss and Japanese Pharmacopoeias, which are essentially the same, except that the residue of codein is weighed. In no case did we find more than 1.29 percent codein sulphate by the gravimetric methods, nor more than 0.94 percent by the volumetric method. This, however, in no way controverts the statements that some of the morphine on the market contains larger quantities of codein, as all our samples were from one source.

Estimation of Morphine in Tablets.—During the past few years, a number of solvents and mixtures of solvents have been proposed for the extraction of morphine in the assay of morphine preparations. For example, some time ago Thorburn[1] proposed the use of phenyl ethyl alcohol as a suitable solvent for this purpose. Engelhardt[2] has recently proposed a mixture of isobutyl alcohol and

[1] Journal Industrial and Engineering Chemistry, October, 1911.

[2] Deutsche Americanische Apotheker Zeitung, 1913.

chloroform for this purpose. The method which has given the best results in our hands, however, is that of Bernegau and Heidlberg, published in last year's Proceedings. We have slightly modified the method as follows:

Dissolve sample equivalent to 0.2 gm. or less of morphine in not more than 15 cc. of water (insoluble matter does not cause emulsions) in a separator, add 50 cc. of amyl alcohol, make alkaline with ammonia and heat on steam bath for 10 minutes, shake for 5 minutes and allow to stand until cold. Draw off aqueous layer into a second separator and pour the amyl alcohol into a 300 cc. Erlenmeyer flask containing a few grains of sand. Repeat the extraction with amyl alcohol twice. Distill off the united amyl alcohol solutions in an oil bath just to dryness. (Do not overheat). Blow out the vapors of amyl alcohol and dissolve the residue of morphine alkaloid in 20 cc. of N/20 sulphuric acid with the aid of chloroform and heat. Titrate back with N/50 potassium hydroxide, using methyl red as indicator.

In a series of experiments, while we obtained from 0.5 to 0.76 percent more than the theoretical quantity of morphine sulphate by the amyl alcohol method, we obtained from 5 to 13 percent less than the theoretical by the phenyl ethyl alcohol method, and from 4.7 to 7.5 percent less than the theoretical by the isobutyl alcohol method.

ANALYTICAL LABORATORY OF H. K. MULFORD COMPANY, June 20, 1913.

OREGON AND CANADA BALSAM OF FIR.*

J. G. ROBERTS AND M. M. BECKER.

Considerable difficulty has been experienced in the past year or two in obtaining Canada Balsam of Fir (Terebinthina Canadensis). It is stated that it is practically unobtainable at this time and that there will be none available until the next crop has been gathered. In view of this fact, it has become necessary to find a suitable substitute. Accordingly there is considerable Oregon Balsam Fir now being offered to the trade. This is an allied natural product and bears a close resemblance to the better known Canada Balsam of Fir.

As information regarding Oregon Balsam of Fir is exceedingly meagre it became necessary in order to obtain data that would assist in establishing the identity and purity of given samples to obtain some Oregon Balsam of Fir from a known source. Such a sample was procured through the courtesy of Mr. R. G. Bailey, who guaranteed that it was a genuine sample of Oregon Balsam of Fir. It is very similar in color, odor and taste to the Canada Balsam but it is noticeably thinner.

We have examined several lots of Oregon Balsam of Fir that were purchased on the open market and have noted several points of difference between them and

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