

have been settled, the work will not be likely to cause much discussion as far as the Chairman is able to judge. The convention settled the question, very largely, of the nomenclature of the new Pharmacopoeia. The recommendation was as follows:

"We recommend that changes in the titles of articles at present official be made only for the purpose of insuring greater accuracy, brevity, or safety in dispensing, and to eliminate therapeutically suggestive titles. In the case of newly admitted articles, it is recommended that such titles be chosen as are in harmony with general usage and convenient for prescribing, but in the case of chemicals of a definite composition the scientific name should be given at least as a synonym.

"There should also be inserted, after each article used by physicians in prescriptions, a carefully considered, abbreviated name, which may be known as an official abbreviation, in order that uniformity may be established throughout the country, with the object of preventing mistakes in reading and compounding prescriptions, and further, to serve as authorized abbreviations in labeling the store furniture of the pharmacist."

This sub-committee has always reported in previous revisions after the other sub-committees have sent their reports and the admissions and deletions have been finally determined.

It is not surprising in Pharmacopoeia work to hear criticisms in certain quarters asking definite information and date for the appearance of the new book. While every effort should be made persistently and continuously to push the work, great patience is required in order that hasty conclusions or incorrect guesses be eliminated. At present, thousands of interested observers throughout the country where there were hundreds before will scrutinize the pages with the utmost care and with very good reason, for the Food and Drugs Act decisions are based upon the standards of the United States Pharmacopoeia, and it is only by continual vigilance that errors may be eliminated and that a work involving so much responsibility can be successfully produced.

Now is the time to send the Chairman suggestions, criticisms and comments in order that they may be thoroughly considered.

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## COOPERATIVE WORK ON A UNIFORM METHOD FOR ALCOHOL DETERMINATIONS.\*

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L. HENRY BERNEGAU.

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At one of our last meetings in 1910 a discussion arose in regard to determining the percentage of alcohol in products and preparations such as Wines, Elixirs, Fluidextracts, etc. At this meeting I stated that the percentage of alcohol determined in a certain N. F. preparation by different chemists varied about 3 per cent. The samples of said preparation were assayed for alcohol by three

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\*Report of Committee of the Scientific Section of the Philadelphia Branch of the American Pharmaceutical Association.

chemists of commercial laboratories, outside of Philadelphia, all of them being known to be highly efficient and reliable.

At the meeting mentioned above it was suggested that samples of a wine and an elixir be submitted to all Philadelphia chemists who were willing to cooperate with us in work on this very important subject. We furnished the samples, which consisted of a wine and an elixir, the latter being made up by myself, and containing only alcohol, water, sugar and caramel. On sending the samples to our chairman, W. A. Pearson, it was stated that the light colored sample was a natural wine, while the darker colored sample was an elixir, containing no highly volatile substances such as ether, chloroform, volatile oils, acids, etc. W. A. Pearson kindly distributed the samples among the Philadelphia chemists who expressed their willingness to cooperate. To each and all of them I would extend my heartiest thanks for their work and interest shown in this subject. Following are the names of the ten gentlemen who received the samples and reported their results:

1. M. Becker, with Smith, Kline & French.
2. C. S. Brinton, Food and Drug Inspection Laboratory, Philadelphia.
3. Geo. E'We, with H. K. Mulford Co.
4. L. Criesmer, a second-year-student at P. C. P., through Prof. LaWall.
5. W. Hilts, of U. S. Food and Drug Inspection Laboratory, Philadelphia.
16. Professor C. H. LaWall.
7. H. B. Mead, Laboratory of Prof. LaWall.
8. C. Roberts, of U. S. Custom House, Philadelphia.
9. H. M. Sechler, with Smith, Kline & French.
16. Otto Stockinger, with H. K. Mulford Co.

The alcohol in the distillate was determined by the different chemists either by means of the pycnometer or by the Westphal balance. The use of the pycnometer is, without doubt, preferable in very accurate scientific work; nevertheless the Westphal balance gives good results if properly handled, and provided the balance is very sensitive to the fourth decimal and standing on a perfectly level plane. Some of the chemists brought the temperature up or down to 15.6° C. before taking the weight or specific gravity, while the others made no corrections in regard to the temperature. Although the temperature has to be taken into account in determinations by any method, it is not necessary for determinations by the Westphal balance to bring the distillate to 15.6° C. It is only necessary to take the specific gravity at the same temperature at which the sample was originally measured. Then by use of the temperature corrections in the alcohol tables, sufficiently accurate results can be obtained. We certainly would obtain bad results if the respective preparations were measured for distillation at about 10° C. and the distillate measured at 35° C. or so. Some of the chemists used calcium carbonate, some sodium bicarbonate, and still others potassium hydroxide for neutralizing the free acids. In some cases the reports did not specify what had been used, or if anything had been used for neutralization.

The results found by the different operators were quite concordant, there being only a difference between extremes of 1.06 per cent. in the wine and 0.78

per cent. in the elixir. If the same method had been used by all, I am sure that much closer results would have been obtained.

The following results were obtained: (I give the names in alphabetical order).

WINE (the light-colored liquid)—

1—M. Becker .....	17.3%
2—C. S. Brinton, 17.71%—17.73%, average.....	17.72%
3—Geo. E'We, 18.22%—18.4%, average.....	18.30%
4—L. Griesmer, 18.36% .....	18.36%
5—W. Hiltz, 17.66%—17.68%, average.....	17.67%
6—Prof. LaWall, 18.28%—18.12%, average.....	18.20%
7—H. B. Meade .....	17.99%
8—C. Roberts .....	17.39%
9—H. M. Sechler .....	17.50%
10—O. Stockinger, 17.65%—17.4%, average.....	17.525%

Mr. O. Stockinger made three determinations of this same wine previous to the sample submitted by W. A. Pearson, and found:

1st—by plain distillation.....	17.6%
2d—after neutralizing with KOH.....	17.8%
3d—with an excess of KOH.....	17.5%
Average .....	17.63%

ELIXIR (the dark-colored liquid)—

1—M. Becker .....	25.00%
2—C. S. Brinton, 25.54%—25.55%, average.....	25.55%
3—Geo. E'We, 25.3% —25.91%, average.....	25.635%
4—L. Greismer .....	25.57%
5—W. Hiltz, 25.55%—25.55%, average.....	25.55%
6—Prof. LaWall, 25.83%—25.7%, average.....	25.765%
7—H. B. Meade .....	25.35%
8—C. Roberts .....	25.35%
9—H. M. Sechler .....	25.54%
10—O. Stockinger, 25.4% —25.4%, average.....	25.4%

In calculating the alcohol percentage from specific gravity by means of the pycnometer or Westphal balance the following tables were used:

Messrs. Brinton and Hiltz used U. S. P. table and also table given in Government Bulletin No. 107 of the Bureau of Chemistry.

Messrs. E'We and Stockinger used U. S. P. table.

Prof. LaWall used U. S. P. table and also table according to Hehner, given in "Leach's Food Inspection and Analysis."

Messrs. Becker, Griesmer, Meade, Roberts and Sechler did not specify which table they used. Most probably the U. S. P. table, as otherwise they would have made some remarks in their reports about it. The difference in these three tables can be called negligible in this case, i. e., in determining the alcohol percentage in wines, elixirs, fluidextracts, etc. The specific gravity by all of the cooperators was taken at 15.6° C. or 60° F., so that no temperature corrections were necessary except by Messrs. E'We, Griesmer and Stockinger, who took the specific gravity at room temperature and made corrections by U. S. P. table. On using the Westphal balance, I personally prefer to bring the distillate to room temperature, taking the temperature exactly during weighing and making corrections afterwards according to U. S. P. table. I would now ask the following questions:

1. What method is preferable in determining the alcohol percentage in this

class of products and preparations (wines, elixirs, etc.), the pycnometer or Westphal balance? I ask the question from the standpoint of a large manufacturing house which sometimes has to make as high as thirty alcohol determinations per day.

2. If the Westphal balance is used, is it preferable to weigh or to take the specific gravity at 15.6° C., or to make temperature corrections according to U. S. P. table?

3. Which of the three tables mentioned is the most nearly correct and which should be adopted for standard: (1) U. S. P., (2) Bulletin No. 107, (3) Leach-Hehner?

4. Which chemical is best suited to neutralize acids with—potassium or sodium hydroxide, calcium carbonate, sodium bicarbonate, etc.?

These four questions refer to the reports of the cooperators' work. In addition, I would ask:

1. Which chemical is best suited to neutralize ammonia—sulphuric acid, phosphoric acid, etc.?

2. What is the best to prevent frothing?

3. What is the best to prevent bumping?

In our work to prevent bumping we use small pieces of unglazed or porous porcelain. To prevent frothing we use with advantage potassium bisulphate, in preparations which contain no acids which, by coming into contact with the acid salt would evolve volatile acids. A layer of melted paraffin is also of value in all cases.

The two samples tested by the cooperators and on which much concordant results were obtained were very simple in regard to their composition. I would greatly appreciate it if the chairman would request the further cooperation of those who assisted with this work, on more difficult problems; that is, on preparations of a more complex nature. I take the liberty of making some suggestions and comments:

A certain class of tinctures, fluidextracts, etc., seem to have an attraction for alcohol and show their unwillingness to part from it by foaming, frothing, bumping, "racketing," etc. Below are given a few of these:

Wild Yam  
Quillaja  
Burdock

Senna  
Asafetida  
Sarsaparilla

Trillium  
Buchu  
Cubeb

From these I would ask that you take your choice for the next series of cooperative tests on alcohol determinations. Some preparations need a double distillation, such as Elixir and Tincture of Ammonia, Aromatic, etc., first by means of sulphuric acid, and second, by means of potassium hydroxide. Preparations containing free iodine should be distilled with sodium thiosulphate and a little potassium hydroxide.

Spirit of camphor, etc., must first be freed from the camphor by the well-known sodium chloride method, before distillation. The addition of calcium hydroxide is of advantage in the distillation of Buchu and Valerian prepara-

tions. Not only will a perfectly clear distillate be obtained, but the bumping will also be considerably diminished.

On distilling unknown preparations with alkalis, tannin should always be used to precipitate any volatile alkaloids.

On closing my report, I would extend my thanks to Mr. Otto Stockinger and Mr. Geo. E'Ve for their valuable help and suggestions.

## SUMMARY.

Chemist	Wine	Elixir	Pycno- meter	Westphal % Plain	Neutral %	Alkaline %	Average %
M. Becker .....	—	—	—	—	—	—	17.3
" .....	—	—	—	—	—	—	25.0
C. S. Brinton.....	—	—	—	—	—	—	17.72
" .....	—	—	—	—	—	—	25.55
G. E'Ve .....	—	—	—	—	18.4	—	18.31
" .....	—	—	—	—	25.3	—	25.635
L. Griesmer .....	—	?	?	—	—	—	18.36
" .....	—	?	?	—	—	—	25.57
M. Hilts .....	—	—	—	—	—	—	17.67
" .....	—	—	—	—	—	—	25.55
Prof. LaWall .....	—	?	?	—	—	—	18.28
" .....	—	?	?	—	—	—	18.12
H. B. Meade .....	—	?	?	—	—	—	25.83
" .....	—	?	?	—	—	—	25.70
C. Roberts .....	—	—	—	—	—	—	17.90
" .....	—	—	—	—	—	—	25.78
H. M. Sechler.....	—	—	—	—	—	—	17.39
" .....	—	—	—	—	—	—	25.35
O. Stockinger .....	—	—	—	—	—	—	17.50
" .....	—	—	—	—	—	—	25.54
" .....	—	—	—	17.6	—	—	17.65
" .....	—	—	—	—	—	—	17.4
" .....	—	—	—	—	—	—	15.5
" .....	—	—	—	—	—	—	25.4
" .....	—	—	—	—	—	—	25.4

ANALYTIC LABORATORIES, H. K. MULFORD, Co., March 12, 1911.

### APPARATUS FOR THE DISTILLATION OF ALCOHOL IN PHARMACEUTICAL PREPARATIONS.

M. BECKER.

In order to overcome the principal physical differences encountered in the estimation of alcohol in preparations the apparatus shown in the accompanying illustration was devised. By its use the need for redistillation due to frothing and bumping is eliminated, consequently saving considerable time and labor.

NOTE.—Since Mr. H. L. Bernegau presented his contribution, no systematic co-operative work has been made by the committee, but Mr. M. Becker, in the Analytic Laboratory of Smith, Kline and French Co., has designed a suitable apparatus for the distillation of most pharmaceutical preparations.