in every instance, whereas he failed on perhaps ten occasions to obtain concordant results when using the process as it appears in the ninth revision of the Pharmacopoeia.

It was therefore suggested by Dr. Lentz that the U. S. P. process be modified, to read as follows:

Heat about 0.5 Gm., accurately weighed, in a mixture of 15 mils of sulphuric acid and 10 mils of nitric acid, over a small flame until the oxides of nitrogen fumes cease to be evolved and the liquid is of a pale yellow color. Cool the mixture, dilute it with 150 mils of distilled water, add 30 mils of solution of hydrogen dioxide and mix well. Now add 5 Gm. of sodium chloride, dissolved in 20 mils of distilled water, then add gradually, with constant stirring, 5 mils of diluted hypophosphorous acid. Stir thoroughly, then allow the mixture to stand until the precipitate has subsided. Filter and wash the precipitate with distilled water. Transfer the precipitate and filter to a flask, add 50 mils of tenth-normal iodine volumetric solution and 2 Gm. of potassium iodide, agitate the mixture until all the precipitate has dissolved, then titrate the excess of tenth-normal iodine volumetric solution with tenth-normal sodium thiosulphate volumetric solution.

DRUG TOPICS.*

No. 3. Evaluation of Wintergreen Leaves.[1]

BY R. W. ADAMS.

The leaves of *Gaultheria procumbens* have possibly never been regarded as an important drug. To-day the average drug clerk of our cities may not know it. If found at all in a drug store it is most likely contained in compressed packages the inside of which is rarely, if ever, seen by the salesman behind the counter.

Wintergreen oil has been replaced for the most part by sweet birch oil and artificial methyl salicylate. But, although the production of true wintergreen oil from the leaves of *Gaultheria procumbens* is still an agricultural or, more correctly, a forest industry of some local importance in certain states, this fact adds nothing to the significance of the drug as such.

Nevertheless wintergreen is still an article of commerce and the person who has occasion to purchase a bale ought to know whether the price asked for the drug is at all indicative of its quality. Drug brokers may judge the leaves by their appearance, yet the best appearing leaves may be valueless so far as available volatile oil is concerned. The writer has even met persons who thought that a nice-looking green leaf was of first quality because the drug had the peculiar wintergreen odor. In most cases, there is reason to suspect that neither collector, broker, nor purchaser cares a rap about the quality of the leaf so long as the price admits of a satisfactory business transaction. The writer knows of at least one bale of about 150 pounds of North Carolina leaf that was sold by a "reliable" firm for a fair price, when the leaves were almost useless for the purpose for which they had been purchased. No doubt the leaves had passed through various hands, yet no one seems to have given any thought whatever to the quality of the leaves. Even though wintergreen leaves are not used where a question of life and death is involved, the mere matter of business honesty seems to demand more consideration than has

* From the Laboratory of Edward Kremers.

[1] Extracts from R. W. Adams, "Oil of Wintergreen from Gaultheria." Thesis University of Wisconsin, 1910.

been given to this drug. If nothing more were accomplished than to drive the ignorant drug collector and vendor out of business something worth while would be accomplished that must react favorably on the entire drug trade.

The commercial value of gaultheria leaves depends on their gaultherin content. The amount of glucoside can readily be ascertained by the colorimetric determination of the methyl salicylate, produced upon hydrolysis, with ferric chloride. As a standard a one-tenth percent solution of artificial ester may be used for comparison.

The conditions most favorable to an assay were ascertained by Ziegelmann[1] who found that for small quantities, such as are used in an assay, 24 hours of maceration at room temperature are the most favorable. These experiments were repeated by Adams who substantiated them. He reports the following results for 6 Gm. lots per assay:

Time of maceration.	Yield.
12 hours	0.63 p. c.
12 hours	0.64 p. c.
24 hours	1.00 p. c.
24 hours	0.97 p. c.
48 hours	1.00 p. c.

Inasmuch as the highest yield of wintergreen oil from gaultheria leaves recorded is only 0.80 p. c., the assay results seem rather high. Indeed, Ziegelmann did not succeed in obtaining, upon distillation on a semi-economic scale, much more than one-half of the oil indicated by assay. His results with sweet birch, likewise, fell materially below the yield indicated by assay. However, he does not appear to have cohobated the aqueous distillates, a fact that may readily account for the low yield of oil. For this purpose the oil distillation experiments were repeated with results tabulated below. The leaves, covered with water, were allowed to macerate 24 hours previous to distillation.

No. of expt.	Wt. of herb.	Wt. of oil.	P. c. oil.	$d_{25}°$.
1.....	48 lbs.	265 Gm.	1.22 p. c.	1.1780
2.....	77 lbs.	492 Gm.	1.40 p. c.	1.1780
3.....	113 lbs.	569 Gm.	1.11 p. c.	1.1730
4.....	140 lbs.	200 Gm.	0.31 p. c.	1.1724

Materials Nos. 1, 2 and 3 had been collected by Indians near Black River Falls, Wis., under the supervision of the Missionary. No. 1 had been collected in the fall of 1909 and thoroughly dried; No. 2 in the spring of 1910 and thoroughly dried; No. 3 also in the spring of 1910, but not thoroughly dried. The yields are high and if the cohobation had been carried farther might, no doubt, have been increased at least slightly. It might be concluded that the leaves collected in spring yield more oil than those collected in fall, but to draw such a conclusion from single experiments would be rash indeed. More striking is the lower yield obtained from leaves collected during the same season but not thoroughly dried (contrast No. 3 with No. 2). The very fact that the leaves had a wintergreen odor, though their appearance was perfect, revealed in itself insufficient drying (due to rainy weather), as is further substantiated by the lower yield of oil.

But, if the Indians failed in one instance to turn out a perfect drug, what

[1] E. F. Ziegelmann, "Oils of Wintergreen and Birch," *Pharm. Rev.*, 23, p. 83.

will one say of the bale of 140 lbs. (No. 4) obtained from a southern state? Here the yield proved to be less than 25 percent of the best yielding drug collected and cured by the Indians (compare No. 4 with No. 2).

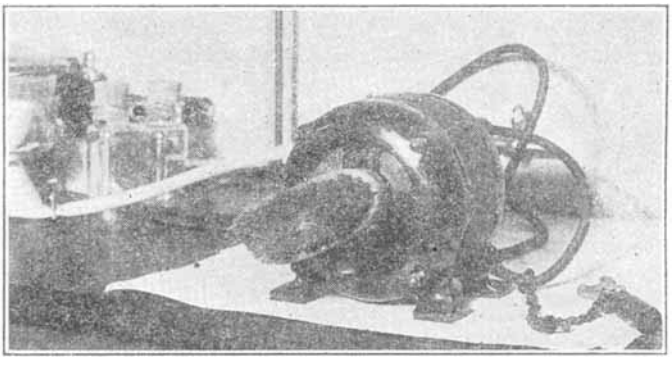
It is further noteworthy that the leaves which had not been properly cured yielded oils with a slightly lower density than that of the oils from perfect leaves.

To claim that the assay of gaultheria leaves yields perfect results would be foolish. As yet we have had too little experience therewith. However, this much may be claimed for it, *viz.*, that it would have prevented such a fraud as the sale of the 140-lb. bale of grossly inferior leaves. More than that, its application to the wintergreen oil industry may yet place the distillation of true wintergreen oil and sweet birch oil on a scientific basis.

THE ASH YIELD OF CALUMBA.

BY E. L. NEWCOMB, C. H. ROGERS AND C. V. NETZ.

Calumba is a drug which is ordinarily considered to be quite clean. A careful study of a series of samples indicates that there may be nearly five percent of dirt present. The majority of commercial samples appear to contain less than one percent of sand, clay or other foreign inorganic matter measured as acid-insoluble ash.



Motor with brush attached—used for preparing *clean* drugs for normal ash determinations.

Carefully cleaned samples of commercial Calumba usually yield between four and five and one-half percent of total ash. The amount of the ash insoluble in 5% hydrochloric acid runs less than one-half percent with the well-cleaned samples.

A method was previously described for removing on a commercial scale the sand and clay from crude vegetable drugs. In working with small quantities of drugs, such as roots, it is not practicable to use a mechanically operated gyrator sifter. For making fairly accurate determinations of the total and acid-insoluble ash of vegetable drugs freed from all foreign matter, one should preferably begin the work with the living material. With many of our drugs from the tropics, this is, however, quite out of the question. The following method is applicable to quite a number of drugs; we have used it successfully and present it for what value it may be to others.