

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).

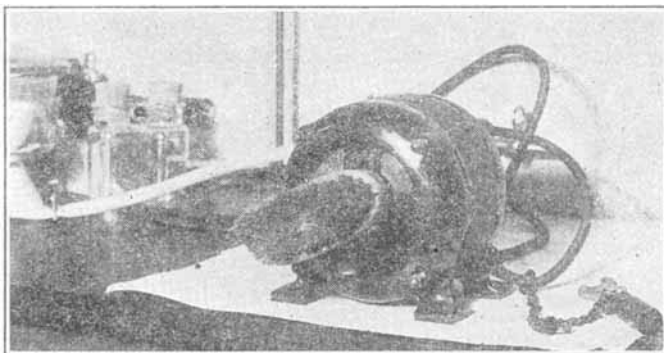
It is further noteworthy that the leaves which had not been properly cured yielded oils with a slightly lower density than that of the oils from perfect leaves.

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.

Motor Brush for Cleaning Drugs.

A small brush, such as the ordinary hand scrub brush, is fastened to the end of the shaft of an electric motor. This may easily be done by making a tapering hole in the center of the back of the brush. The brush may then be wedged on to the shaft. The motor which we have used is a General Electric Co. machine— $\frac{1}{4}$  H. P., 1730 R. P. M., alternating current. This machine may be connected to almost any electric light line with alternating current. The switch is turned on and the selected pieces of the drug held firmly but gently against the rapidly revolving brush. A little practice is necessary in order to simply remove the fine particles of foreign matter and not to burnish the sample. With a drug like Calumba, for example, it is important not to remove any of the corky layer, on account of the stone cells containing calcium oxalate which occur in this tissue. With proper care, the method will yield as near perfectly clean samples as we may expect.

The following results relate to the standard for Calumba:

TOTAL AND ACID-INSOLUBLE ASH OF CALUMBA. BY C. H. ROGERS AND C. V. NETZ.

| Sample No. | Source of sample and remarks.  | Percent total ash. | Percent insoluble in 5% HCl. |
|------------|--|--------------------|------------------------------|
| 1.         | Commercial powder, bought 1920.....  | 6.66               | 0.89                         |
|            |  | 6.48               | 1.06                         |
| 2.         | Commercial powder, bought 1910.....  | 9.43               | 4.26                         |
|            |  | 9.27               | 4.54                         |
| 3.         | Commercial powder, bought 1920 (labeled "3.32% ash").....  | 8.04               | 3.49                         |
|            |  | 8.02               | 3.29                         |
| 4.         | Commercial sample, bought 1908, powder.....  | 6.64               | 1.46                         |
|            |  | 6.77               | 1.62                         |
| 5.         | Commercial whole drug, powdered in this laboratory 12/4/20 without cleaning.....                         | 5.64               | 0.72                         |
|            |  | 5.61               | 0.78                         |
| 6.         | Commercial whole drug, bought 1920, powdered in this laboratory 12/4/20 without cleaning, No. 30.....    | 6.43               | 1.35                         |
|            |  | 6.53               | 1.38                         |
| 7.         | Commercial whole drug, bought 1920, powdered in this laboratory 12/4/20 without cleaning, No. 30.....    | 4.76               | 0.35                         |
|            |  | 4.72               | 0.38                         |
| 8.         | Commercial whole drug, bought 1920, powdered in this laboratory 12/4/20 without cleaning, No. 30.....    | 6.31               | 0.67                         |
|            |  | 6.39               | 0.57                         |
| 9.         | Commercial whole drug, bought 1920, powdered in this laboratory 12/4/20 without cleaning, No. 30.....    | 5.74               | 0.82                         |
|            |  | 5.86               | 0.78                         |
| 10.        | Commercial sample, whole drug, bought 1920, powdered in this laboratory 12/4/20 without cleaning.....    | 5.12               | 0.60                         |
|            |  | 5.13               | 0.51                         |
| 11.        | Commercial whole drug, same as No. 5, above, except cleaned by motor brushing and powdered, No. 30.....  | 5.16               | 0.49                         |
|            |  | 5.41               | 0.43                         |
| 12.        | Commercial whole drug, same as No. 10, above, except cleaned by motor brushing and powdered, No. 30..... | 5.42               | 0.38                         |
|            |  | 5.45               | 0.30                         |
| 13.        | Commercial whole drug, same as No. 7, above, except cleaned by motor brushing and powdered, No. 30.....  | 4.31               | 0.31                         |
|            |  | 4.29               | 0.29                         |
| 14.        | Commercial whole drug, same as No. 6, above, except cleaned by motor brushing and powdered, No. 30.....  | 4.35               | 0.32                         |
|            |  | 4.48               | 0.20                         |

|   |      |      |
|---|------|------|
| 15. Commercial whole drug, same as No. 9, above, except cleaned by motor brushing and powdered, No. 30..... | 4.13 | 0.42 |
|   | 4.17 | 0.47 |
| 16. Commercial whole drug, same as No. 8, above, except cleaned by motor brushing and powdered, No. 30..... | 3.74 | 0.33 |
|   | 3.76 | 0.35 |

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## THE VOLATILIZATION OF ETHYL NITRITE FROM SPIRIT OF NITROUS ETHER.\*

BY J. G. ROBERTS.

An investigation to determine the cause of the deterioration of spirit of nitrous ether was instituted on account of the frequent prevalence of low strength samples which had, supposedly, been stored under normal conditions.

As shown by the following results deterioration is due to either the decomposition of, or to the volatilization of the ethyl nitrite. Decomposition is due to the action of light and volatilization to the action of heat, to the imperfect sealing of containers or to insufficiently filled containers. Carelessness or slowness in mixing the ethyl nitrite and alcohol is also a contributing factor. The latter cause can, however, be eliminated, particularly when a small quantity is prepared, by using the sealed tubes which contain sufficient ethyl nitrite, which when mixed with one pint of alcohol will produce spirit of nitrous ether of official strength.

Ethyl nitrite or nitrous ether as it was first named is a well-established product which has been well known for a considerable period. It was discovered by Kunkel as early as 1681 and was obtained by the reaction of nitric acid, alcohol and copper. It is a yellowish volatile liquid of a pleasant, ethereal odor, has a specific gravity of 0.990 at 15.5° C. and boils at 17° C. It is readily miscible with alcohol from which it is easily dissipated when not properly stored.

In the present method of manufacture, ethyl nitrite is produced by the reaction of sodium nitrite, sulphuric acid and alcohol. It is preferably made in a stoneware vessel of convenient size which is provided with a mechanical stirrer. The resulting gaseous ethyl nitrite is passed through a well-cooled condenser and collected in an ice-packed receiving vessel. As it is decomposed in the presence of water, particular care is taken to render it anhydrous.

Spirit of nitrous ether is a very popular article and judging from the attention given it, it has the greatest popularity among the various national, state and municipal authorities, who have made it a very frequent cause of investigation and examination. That their vigilance is justified is proven by the large number of cases of low quality sweet spirit of nitre that they have found. The ready volatility of its ethyl nitrite content has always been a disturbing factor and is a matter of deep concern to all those who take pride in the quality of the preparations they dispense.

According to modern chemical classification ethyl nitrite is an ester and not an ether. Its ethereal quality was probably the reason for the name or possibly on account of the discarded, inaccurate term "compound ether" that was formerly

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