THE PREPARATION OF THE OAK TANNINS, WITH SPECIAL REFERENCE TO THE USE OF ACETONE AS A SOLVENT.[1]

BY HENRY TRIMBLE AND JOSIAH C. PEACOCK.

THE usual method for preparing a tannin from a substance as rich as nutgalls, or containing from sixty to seventy per cent. of the astringent principle, is to extract with a mixture of alcohol and ether, or, what amounts to the same thing, official ether, sp. gr. 0.750. When, however, the material is an oak bark, containing from four to fifteen per cent. of tannin, the choice of a proper solvent becomes a more difficult matter.

During the past year a number of experiments have been made on oak bark with a view of determining the most satisfactory solvent for the tannin. The following are especially worthy of consideration:

(1) Official ether sp. gr. 0.750, which is equivalent to a mixture of alcohol and ether.

(2) Acetic ether.

(3) Water.

(4) Acetone.

The greatest objections to ether are its expense and the slowness of its solvent action, which consume time as well as a large amount of menstruum.

Acetic ether is a much better solvent, and the expense is the chief difficulty in the way of its use.

Water is slow in its solvent action; this, however, is in part overcome by long maceration, and then slow percolation. The tannin must be separated from the resulting aqueous solution, either by agitation with acetic ether, or by precipitation with lead acetate. In the latter process it was found possible at a considerable sacrifice of oak bark to procure a quantity of light colored tannin, by precipitating one-half of the aqueous percolate with lead acetate, collecting the precipitate, stirring it through the other half of the percolate, and then filtering. The

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filtrate was very light in color, and was either evaporated under reduced pressure and submitted to further purification to be described hereafter, or it was agitated with acetic ether, and, after removal of the latter solvent, purified in the same manner.

Apart from the slowness of the process, the yield of tannin after purification was always small when water was used as a solvent.

Within the past few years acetone has appeared in commerce in a nearly pure form. Its solvent action has been suggested for several plant principles, but thus far little, if any, reference has been made to its use as a solvent for tannin, although there is good reason for believing that some manufacturers are using it for the extraction of nutgalls. It is cheaper than ether, but more expensive than alcohol. It is a better solvent of tannin than either of these, and extracts the tannin with less sugar and other carbohydrates, because of its poor solvent power over these. Its low boiling point, 56.5°, renders its recovery easy and rapid, without danger of decomposition to the tannin.

From a sample of powdered nutgalls, commercial ether extracted 59.77 per cent. of solids, while acetone extracted 62.24 per cent. of the same.

The following process, after some preliminary experiments, has been devised and thus far proven satisfactory:

The powdered oak bark was well moistened with acetone, packed in a glass percolator, and the menstruum poured on until it commenced to drop from the lower orifice, when the latter was closed with a cork, and the bark allowed to macerate for forty-eight hours. Enough of the solvent was poured on before maceration commenced to keep a thin layer of it above the drug. A glass plate smeared with petrolatum was kept on top the percolator to prevent evaporation. At the expiration of the maceration period, the stopper was removed and the percolation continued rapidly until the number of liters of percolate amounted to one-half the number of kilograms of oak bark used. The latter was then usually found to have been exhausted. In some instances a No. 20, in others a No. 40 powder, was used. In every case the acetone rapidly penetrated the drug and accomplished complete exhaustion.

The acetone was removed by distillation, the first portion on a water bath, under ordinary conditions, but the last portions by the additional aid of reduced pressure. The residual product was a dark red or brown semi-solid extract. This was warmed with water until nearly all of it dissolved. After cooling the whole was filtered and the clear filtrate was diluted with water so long as precipitation took place. This dilution separated much of the anhydrides. The filtrate from these was of a clear red color and yielded no further precipitate on the addition of water. It was then agitated successively with acetic ether. The acetic ether portions were mixed and the solvent recovered by distillation under reduced pressure, which yielded the tannin in a porous or "puffed up" condition. The product was then treated with cold water, and, after filtration, was again separated by agitation with acetic ether. This process was continually repeated until the tannin was readily and completely soluble in water. The tannin then possessed considerable odor of acetic ether, which was removed by solution in official ether, sp. gr. 0.750, and, after filtering clear, distilling off the solvent under reduced pressure. The product was then digested with absolute ether, which dissolved the small amounts of adhering resin and crystalline principles which occur along with it in the bark or result from decomposition when working it, and the tannin remained behind nearly pure, and readily and completely soluble in water.

This process was carried out on barks from the following species of oaks: *Quercus alba*, *Q. coccinea* and its variety *tinctoria*, *Q. falcata*, *Q. palustris*, *Q. Prinus*, *Q. bicolor*, *Q. stellata*, *Q. Phellos*, and *Q. rubra*.

It was found in some cases that by dissolving the acetone residue in a mixture of four parts water and one part alcohol, instead of water alone, there was less formation of anhydrides.

A few trials were made with a modification of the purification process in which the first acetic ether residue was dissolved in water and filtered through a freshly prepared lead compound obtained by precipitating a portion of the aqueous solution of the bark with lead acetate.

In some instances the resulting filtrate was nearly colorless, but the loss of tannin was such as not to warrant the adoption of the process for general use. It might, however, be applied in certain cases with satisfactory results. From the colorless filtrate the tannin should be removed by agitation with acetic ether, and the remainder of the general purification process then carried out.

MILK, SKIM MILK, AND WHEY; A STUDY OF THEIR COMPARATIVE COMPOSITION AND SPECIFIC GRAVITY.

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THAT the specific gravity of a milk is dependent upon the composition of the milk and varies according to the per cent. of each constituent is generally admitted. Upon this fact is based Richmond's extended formula. Having given the specific gravity, total solids, and ash of a sample of milk it is possible to calculate by means of this formula the percentage of fat, proteids, and sugar.

The formula is as follows: $P = 2.8\,T + 2.5\,A - 3.33\,F - 0.7\frac{G}{D}$ (P = proteids; T = total solids; A = ash; F = fat; D = density, water at 60°F being taken as one; and $G = 1000\,D - 1000$) From this formula Richmond calculates that each gram of proteids in 100 cc. of milk raises the gravity 2.57°; or that the density of the proteids of milk in solution is 1.346.

Dr. Duprè has found by direct experiment that one per cent. of casein raises the specific gravity of milk 2.55°, or that the density of casein in solution is 1.34.

Mr. Hehner by a different method of investigation concludes that the specific gravity of casein in solution is 1.3106.

From this figure as its density in solution, we conclude that each per cent. of casein raises the gravity of milk 2.36°.

I have recently made a comparative study of the specific gravity and composition of milk and the whey obtained therefrom. One of my objects in this work was to obtain data for determining the effect of each per cent. of casein on the specific gravity of milk and the density of casein in solution.

The following table shows the results of my work: