

ing the same turbidity in 90% alcohol as the second sample when tested ten months later.

Inasmuch as the samples pass all other requirements for pure castor oil, it did not seem that the oil could have been adulterated but that the incomplete and slowly increasing insolubility was due to some inner change of the molecule. This is all the more evident when it is remembered that all oils other than castor oil have very low acetyl values, so that even a 5% contamination with other oils would have reduced the acetyl value below 140, whereas both of the samples showed normal values of 148.0 and 146.5, respectively. Further the portions insoluble in the various alcohols as given above were completely soluble in 95% alcohol, thus offering further proof that the insoluble portion was not vegetable oils, but more probably castor oil.

At the suggestion of I. F. Laucks, we obtained further proof of this theory. A large quantity of the oil was treated with the alcohol, and the insoluble portion was allowed to settle out. This insoluble portion was freed completely from alcohol and water and a portion tested for its acetyl value. Inasmuch as all other oils have values of less than 30 for acetyl value, this insoluble fraction would have a very low acetyl value if the oil had been adulterated. The value as determined, however, was 136, proving conclusively that the insoluble portion was hydroxylated and therefore castor oil and not any other vegetable oil. This acetylated residue also showed an iodine value of 79.4, which is about what would be expected.

We therefore believe that failure to pass the solubility tests for castor oil especially those specifying alcohol of less than 95% strength should not be considered as proof of adulteration, but rather as an indication that further chemical investigation of such a sample is needed to prove or disprove its purity.

Further, while all authors state that the filtration and distillation methods of determining the acetic acid liberated from acetylated oil, give the same results, we have been unable to obtain concordant results by the filtration method. The results are always variable and low. Even if the liquid is cooled before filtering, a loss results. This is probably due to the volatilization of the acid. However, distillation, especially if phosphoric acid is used for acidulating instead of sulphuric acid, always gives concordant, and consistent results. This method is used for determining the acetic acid in calcium acetates, and we have found it much better for determining the acetyl value than the use of sulphuric acid. If the liquid being distilled is allowed to concentrate to a volume of 50 cc. or less, the sulphuric acid is liable to char the acetylated oil (particularly that material which spatters the sides of the flask above the liquid), and liberates volatile acids which go into the distillate and vitiate the results. By using phosphoric acid, that danger is obviated and we can obtain consistent results with good checks, whereas by the filtration method we obtained six different values ranging from 125 to 143 on a sample of medicinal castor oil of known purity, and with a true acetyl value of 148 as determined by the distillation method.

Our method is as follows:

Boil gently 20 grs. of filtered dry oil with 20 cc. of acetic anhydride and 10 grams of anhydrous sodium acetate in a round-bottomed 150 cc. flask (attached to an air-jacketed reflux condenser) for two hours, allowing no material to cake on the sides. Cool somewhat and before breaking connections rinse down the condenser with water, adding about 50 to 80 cc. of water. Pour the contents of the flask into 500 cc. of water in a large beaker and boil for 30 minutes, using glass beads or a stirring rod reaching the bottom of the beaker to prevent bumping. Siphon off the water and

boil the oily layer with fresh water until the wash water is no longer acid to litmus. Separate the acetylated fat from the water, filter and dry in an oven at 100°C.

Weigh 2-4 grams of the acetylated fat into a 500 cc. Erlenmeyer flask, add 50 cc. of alcoholic potash (40 grs. to the litre) and saponify for one hour. Evaporate off the alcohol and dissolve the soap in water. Fit up the usual steam distillation apparatus using a two-litre flask for the steam boiler and allow the steam to escape from the flask for 15 minutes to insure the removal of carbon dioxide from the water. Acidulate the soap solution of the saponified acetylated fat with phosphoric acid using methyl orange as an indicator, conduct the steam into this liquid, and keep both the flasks boiling. Catch the distillate from the Erlenmeyer flask in 40 cc. of quarter normal caustic solution, being careful that the caustic solution seals the end of the delivery tube. After 400 cc. have passed over remove the receiver and titrate back with acid. Continue the distillation, it not being necessary to use caustic in the receiver but being sure that the delivery tube is sealed in with water. Test each 100 cc. of distillate until not more than 0.1 cc. of quarter normal caustic is necessary to produce a red color with phenolphthalein.

The total number of ccs. of quarter normal caustic used to titrate the distilled acid multiplied by 2.5 and by 5.61, and divided by the weight of sample taken, gives the acetyl value. It is better to keep the volume of liquid in the Erlenmeyer flask as small as possible during the distillation to prevent too much splashing, with its consequent carrying-over of the acid solution by entrainment.

OCTOBER ADDITIONS TO MEMBERSHIP American Oil Chemists' Society.

ACTIVE.

- A. L. Knisely, 114 Marion St., Seattle, Wash.
- Leroy H. Habenicht, Kershaw Oil Mill, Kershaw, S. C.
- W. C. Lord, Falkenburg, Seattle, Wash.
- P. McG. Shuey, 115 E. Bay street, Savannah, Ga.
- D. L. Weatherhead, Wilson-Martin Co., Philadelphia, Pa.
- Miss Jane E. Mayo, 93 Dodd avenue, Atlanta, Ga.
- L. E. Walter, Armstrong Packing Co., Dallas, Texas.
- Chas. J. Haines, The Electro Co., Peoria, Illinois.
- Landon C. Moore, 1713 Young St., Dallas, Texas.
- Alexander W. Beemer, A. E. Staley Mfg. Co., Decatur, Ill.
- Wm. B. Byers, Southern Cotton Oil Co., Goldsboro, N. C.
- Carl Neutzel, F. S. Royster Guano Co., Baltimore, Md.
- J. W. Schwieger, Southern Cotton Oil Co., Charlotte, N. C.

CORPORATION.

- Barrow-Agee Laboratories, Jackson, Miss.
- Falkenburg & Co., 116 Yesler Way, Seattle, Wash.
- Hazlehurst Oil Mill and Fertilizer Co., Hazlehurst, Miss.
- Larrowe Milling Co., Rossford, Ohio.

CORRECTION

Correction is gladly made of an annoying error in the October issue, where, in the list of Full Certified Referee Chemists, the Fort Worth Laboratories appeared as the "Fort Smith" Laboratories. To all familiar with the names of the chemical laboratories serving the oil mill industry the mistake would have been self-evident.