

Acknowledgments are due Mr. A. H. Low for suggestions in treating the sulphides with sulphuric acid and acid potassium sulphate, while Frederick Ibbotson and Harry Brearley gave the idea of reducing tin solution with powdered antimony, in an article appearing in the Analyst, 1902, 25.

WILSON H. LOW.

CUDAHY PKG. CO.
SOUTH OMAHA, NEB.

[CONTRIBUTION FROM THE BUREAU OF CHEMISTRY, U. S. DEPARTMENT OF AGRICULTURE.]

THE DETERMINATION OF THE SAPONIFICATION NUMBER OF LUBRICATING OILS CONTAINING SAPONIFIABLE FATS.

HERMAN SCHREIBER.
Received November 1, 1906.

In examining lubricating oils, which were mixtures of mineral oil with saponifiable fat, much difficulty was experienced in determining the saponification number.

Saponifications made with alcoholic potash require a long time and tend to give slightly high results due, in part at least, to the etching of the glass. The substitution of a silver flask for glass (as has been proposed) would undoubtedly be an advantage, but if one to three dozen samples are to be examined at one time, the process will be tedious unless a goodly supply of silver is available.

Several solvents of mineral oil were tried, together with alcoholic potash, and it was finally found that a mixture of benzene and alcoholic potash gave very satisfactory results.

About 5 grams of oil were treated with 25 to 50 cc. of approximately half-normal alcoholic potash and 25 cc. of benzene. In case of some heavy cylinder oils more benzene was used with equal success. However, it was not found necessary with any oil so far examined to use more than 50 cc. of benzene to obtain a clear solution when warmed. If one is obliged to use 50 cc. of benzene, it is sometimes necessary to add neutral alcohol to clear the solution.

The oil was weighed into a 200 cc. Erlenmeyer flask, the alcoholic potash and benzene run in, and a rubber stopper, fitted with a three-foot air condenser, tightly inserted in the neck of the flask. The flask was then set on the iron plate, which forms the top of the steam bath, so that the steam would not strike it directly, and the heat so regulated that the condensing liquid would not be forced out of the top of the condenser. In this way the content of the flask can be boiled without apparently losing any of the solvent. One-half hour was found long enough to saponify any oil examined. The benzene does not interfere with the titration in any way. It separates out, leaving the alkali in the lower layer. In making the alcoholic potash 95 per cent., alcohol was used.

In determining the value of the method, the following results were obtained:

Fat.	Methods.		
	I	II	III
Cottonseed oil	201	203.8	202.8
Corn oil	192	193.2	193.4
Rosin oil	76.9	77.4	75.1
Tallow	196.5	196.9	196.5

Each figure represents the average of two determinations.

The number given under method I is the saponification number obtained when the oil was saponified with alcoholic potash only.

The number given under method II is the saponification number obtained when the oil was saponified with alcoholic potash and benzene.

The number given under method III is the saponification number of the oil obtained when it was mixed with an equal quantity or more of mineral oil, and the saponification number determined by means of alcoholic potash and benzene.

In mixing the oils they were weighed into the Erlenmeyer flask, warmed on the steam bath and shaken to mix as thoroughly as possible. The amount of alkali used up by the mineral oil was separately determined and allowance made for it.

The oils were saponified for one-half hour in each case.

THE FULLER'S EARTH TEST FOR CARAMEL IN VINEGAR.

By W. L. DUBOIS.

Received Oct. 27, 1906

The fuller's earth test for caramel appears in a number of publications covering methods for food analysis, and has been used quite generally for the detection of added caramel in cider vinegar. In some cases the method has been published with no statement of the precautions necessary to its manipulation, nor the severe limitations to which it is subject. In order to investigate these points and if possible prescribe conditions under which it could be applied with certainty, the work described in this article was undertaken.

Fifty samples of pure cider vinegar were obtained from farmers in Pennsylvania through the agents of Dr. B. H. Warren. Of these eleven were selected, differing as much as possible in physical appearance. Five vinegars made by the author in 1905 were also included in the experiment.

Samples of fuller's earth were procured from several houses and from a number of food chemists, the purpose for which the samples were desired being stated. The method was applied as follows:

Fifty cubic centimeters of vinegar and 25 g. of fuller's earth were measured out into a 250 cc. beaker, stirred thoroughly and allowed to stand one-half hour. The mixture was then filtered through a dry folded filter, and the color of the filtrate compared with that of the untreated vinegar, filtered in the same way. Color comparisons were made in a Duboscq colorimeter. In the table below the results are expressed as