MUTER'S METHOD FOR THE ANALYSIS OF FATS AND OILS.[1]

BY NATHANIEL J. LANE.

In the *Analyst* of April, 1889, Muter publishes a method of analyzing fats and oils, which consists in determining the percentage of fluid acids and their iodine absorption.

The method is as follows:

It is first necessary to described "Muter's Olein Tube." This is essentially a burette with a bulb at the top, and provided with a well-ground stopper. The graduated part is of such a width as to show distinctly one cc. in the graduations, and it has a stop cock at a height of fifty cc. from the bottom. The graduations commence at forty cc. from the lower end, and continue up to 250 cc., ending just below the bulb. The tube can be fastened in an ordinary clamp, and may be arranged in a water jacket in warm weather.

THE PROCESS.

Weigh off about three grams of the oil or fat in a tared flask; an Erlenmeyer of about 200 cc. capacity is convenient. To this add fifty cc. alcohol and a fragment of solid potassium hydrate sufficiently large to saponify the fat. Close the mouth of the flask with a cork carrying a long tube drawn to a point, and heat on a water bath until completely saponified; add a drop of phenolphthalein and then acetic acid to faintly acid reaction and drop in alcoholic potash until the very *slightest* pink tinge is restored. 200 cc. of boiling distilled water are placed in a 500 cc. basin, thirty cc. of a ten per cent. solution of lead acetate are added and the whole boiled. Into this the soap solution is slowly poured, with constant stirring, and the whole allowed to cool slowly stirring well so as to cause the precipitate to agglomerate, and the clear liquor is poured off. Boiling water is poured on, and the precipitate washed by decantation, working rapidly. When the washing is complete, the precipitate is scraped from the basin into a stoppered bottle, and eighty cc. of *redistilled*

[1] Read before the New York Local Section, June 2, 1893.

ether having been poured on it, the remaining particles of the precipitate are washed from the basin into the bottle with ether, and the bulk of ether in the bottle is made up to 120 cc. The bottle is securely closed and allowed to stand (with occasional shaking), for twelve hours, during which time the lead oleate will entirely dissolve. A funnel with a ground edge is arranged over the "olein tube," a filter is placed therein and the contents of the bottle having been filtered into the tube, the insoluble lead stearate, etc., remaining on the filter, is washed with ether until the washings pass free from lead. This will usually be attained by the use of 100 cc. of ether; during the filtration and washing the funnel must be kept covered by a ground glass plate. The funnel having been removed, dilute hydrochloric acid is poured into the tube up to the first mark (thus using about forty cc. of acid, one in four) and the tube having been closed by the stopper and taken from the clamp, is thoroughly shaken until the decomposition is complete, which is indicated by the ethereal solution clearing up. The tube is returned to the clamp, the liquids allowed to separate, and the aqueous layer is run off by the bottom pinch cock. Water is then poured in up to the same mark, the whole is again shaken and separated as before, and this is repeated until the washings are drawn off free from acidity. Water is then once more run in until the ether is forced up to the zero mark, and ether added (if necessary) so as to bring its upper layer to a definite point (say 200 cc.), and the total volume is read off and noted. Fifty cc. of the ethereal solution of the fluid acids are run into an Erlenmeyer flask, and the flask having been attached to a condenser, the ether is distilled off by a bath of warm water until only a little remains. It is important that the whole of the ether should not be distilled off, so as to avoid contact of the acids with the air. Fifty cc. of pure alcohol (or methylated spirit that has been rectified over potassium hydrate) are added to this residue, and the solution is titrated with decinormal soda,

[1] Since reading this paper, I have noticed an article in the *Analyst* of December, 1892, by Otto Hehner on the separation of solid and liquid acids, in which he says that the accurate separation of the above acids by solution in ether is impossible, owing to the solubility of lead stearate in ether, but as he extracted in a Soxhlet, the ether was, of course more or less warm and I have found that the hot ethereal solution invariably holds up lead salts of solid acids, which are deposited on cooling.

using phenolphthalein as indicator. Each cc. of soda used represents 0.0282 of oleic acid, and the amount found is calculated up to the total bulk of the ethereal solution, so as to obtain the total liquid acids in the weight of fat started with.

Run off as many cc. as will contain 0.5 gram of oleic acid (or as nearly that quantity as can be conveniently measured by the instrument, of course carefully noting the amount taken) into a stoppered bottle, of at least 350 cc. capacity the ether distilled off in a current of carbonic acid, which has been passed through a solution of sodium bicarbonate and dried over calcium chloride, and when *every trace* of ether has been expelled, fifty cc. of Hubl's solution are added, set aside in a perfectly dark place, with a blank and after twelve hours titrated as usual.

While the results obtained by this process were unexceptionable, it was too tedious for factory work; therefore experiments were made to shorten it, which resulted in the following modification: Precipitate the lead soaps in a 600 cc. Erlenmeyer flask, in place of a basin, cool under running water, giving the flask a circular motion, which causes the precipitate to adhere to the sides; when the flask is perfectly cold wash by decantation, using hot water, and rinse once with alcohol, then add 120 cc. of ether (redistilled), attach a reflex condenser and boil on a water bath until all the lead oleate is dissolved and the insoluble portion is a fine powder. Then cool *thoroughly*; this is very important, as some of the lead compounds are soluble in hot ether, but not in cold. The object of boiling the lead soaps with ether is to shorten the filtration, for when treated as Muter directs, the filtration takes six to eight hours, while boiling reduces the time from twenty-five to fifty minutes. The balance of the process is carried out exactly as Muter directs.

The following are some results obtained by the process:

	Sample number.				
	570	571	572	573	574
Liquid acids	68.05	75.16	77.64	76.73	76.67
" " iodine number	141.09	141.96	143.49	141.97	140.59
Hubl's number	99.30	108.36	110.96	108.95	109.229
Saponification equivalent	288.21	297.51	296.27	284.75	288.94
Titer of fatty acids	36.6	32.4	30.4	33.8	33.40

No. 570 is Cotton Seed Stearine, one year old.
" 571 is Summer Yellow C. S. oil, one year old.
" 572 is Winter Yellow C. S. oil, one year old.
" 573 is C. S. oil from Columbia, S. C.
" 574 is C. S. oil from New Orleans.

	Sample number,			
	575	1529	1530	1626
Liquid acids.....	64.15	83.97	86.96	79.84
" " iodine number	99.48	144.40	139.40	114.00
Hubl's number.....	64.96	121.70	122.70	93.53
Saponification equivalent.	287.85	.....	.....	.....
Titer of fatty acids	37.4	.....	.....	.....

No. 575 is an average sample of Steam Lard.
" 1529 is Corn Oil.
" 1530 is " "
" 1626 is Peanut Oil; the solid fatty acids showed Arachidic acid in large quantity.

The iodine number of the liquid lard acids is unusually high; Muter gives ninety-two to ninety-four, but this seems rather too low, the average being about ninety-six. I hope, at some future period to make a more complete investigation of this method, and present the results to the Society.

LABORATORY NOTES.

By Frank Julian.

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DETERMINATION OF CARBON IN STEEL.

After dissolving a sample of wrought steel in copper-ammonium-chloride or copper-potassium-chloride, the carbon remains in the form of a soft mass, which is tedious to filter and troublesome to wash completely. These difficulties may be overcome by the use of the following device: A piece of stiff platinum gauze about fifty mm. in diameter is bent into the form of a shallow dish, and to the edge is soldered a thick platinum wire which hooks over the edge of the beaker, suspending the gauze about forty mm. below. The saturated copper solution is poured on the drillings in the gauze (to remove air-bubbles) until it is covered, and the beaker allowed to stand until complete solution has occurred. This will require from one to four hours, depending on