

ON A MODIFIED FORM OF SOXHLET'S EXTRACTOR.

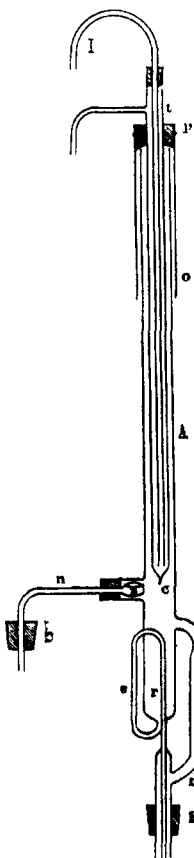
BY WALTER H. KENT, Ph.D.

In the examination of milk in the Health Department of Brooklyn, we have had occasion to try some of the various methods for the determination of fat in milk. In 1885, while the American Society of Public Analysts was endeavoring to fix upon some method, Dr. Otto Grothe and myself made comparative determinations by some of the methods, the results of which have already been published.*

While engaged in this work the form of extractor here described, was devised and first constructed by Dr. Grothe. With some minor improvements, which I have been able to introduce, this extractor has since been used and with much satisfaction.

It will be seen to be a combination of the Soxhlet extractor with an external condenser, as described by Dr. R. Wollny.†

When in use the extractor is attached by a clamp at *A* to a firm support. The support I use consists of an extension of the gas pipe from the table to the ceiling of the room, where it is secured by passing through the wooden ceiling. The condenser, the lower point of which is seen at *c*, rests by the cork *p* on the upper end of the outer tube *A*, the cork being concave below so as to keep the condenser in the center. The condensing tube *t* is firmly joined by the cork *p* to the outer tube *o*, by which the point *c* is also held in the center, if necessary, by the aid



of a little wedge of wood between the tube *A* and the lower end of the tube *o*.

* Annual Report of the Department of Health of Brooklyn, N. Y., 1885, p. 113; London *Analyst*, 1885, p. 47.

† Fresenius Zeitschrift, 1885, p. 106.

In introducing the dried milk into the receptacle *r*,* the clamp at *A* is loosened and the lower part removed, while the condenser is supported at *t* by a string to the support above. As I have it arranged a projecting ring from the gas pipe also serves to support the condenser.

As a means of introducing and withdrawing the tube containing the substance to be extracted, a white thread, previously treated with ether, may be connected with the tube; during the extraction this thread extends over the tube *A* and out under the tube *n*. The tube *n*, for finally withdrawing the ether, is sufficiently large to allow the ether to flow through without obstructing the tube; any ether vapor from the receiving flask at *b* can thereby pass upward into the condenser. The flask serves by the cork *b*, to close the tube *n* during the withdrawal of the ether by the spoon *x*, while the tube *n* also serves the double purpose of supporting the flask by the cork *b*. If the flask at open and supported by other means, the ether vapor from the tube *b* is left *v* passes out at *n*, without being forced up into the condenser, and is thereby lost.

The flasks used hold about 150 c.c. They have a short, wide neck and all fit the same cork. The corks *b* and *h*, being of the same size, the flasks may be used interchangeably. The weighed flask at *b*, serves to close the tube *n* during one extraction, and, after finally receiving the ether, is ready to take the place of the fat receiving flask in the next extraction. A third flask may take the place *b*, and thus another extraction is made while the fat of the first extraction is being dried. In constructing the extractor, the syphon tube *s* should be small, so as to fill readily with ether as it flows over the upper bend of this tube. The vapor tube *v* should be sufficiently large to permit the upward passage of the vapor without being obstructed by the ether that condenses in this tube. A pocket in the tube at the bend *m* should also be avoided.

Of the various materials used as media on which to dry the

* In the drawing the syphon tube *s* extends downward in front of the receptacle *r* of the main tube *A*.

milk, I much prefer asbestos, used as described by Dr. S. M. Babcock.*

The size of the tube for the asbestos may be regulated by the size of the receptacle *r* of the extractor. The perforation in the bottom of the tube should be large enough to allow a rapid discharge of the ether. A tuft of cotton, which has been purified by extraction with ether, covers the hole in the bottom of the tube, over which about two inches of the tube is gently packed with ignited asbestos. Another tuft of cotton, which may be removed for the introduction of the milk, serves to cover the mouth of the tube and to retain the asbestos. The milk may be weighed in the asbestos tube thus prepared, the tube being held upright by the support, with which balances are often provided. During the weighing and drying of the milk it is an advantage to keep the tube upright, as by so doing the milk is more uniformly distributed over the asbestos.

For a holder of the tube while drying, a small beaker may be provided with a doubly perforated, tightly fitting cork. Through one perforation the asbestos tube passes to near the bottom of the beaker, while a tube through the other perforation serves to connect with the pump. Instead of beakers I use cupping glasses; these are stronger and therefor permit the cork to be more tightly fitted by pressure.

I prefer the asbestos method to that using paper, as described by Adams,† and afterwards adopted by the English Society of Public Analysts for the following reasons:

1st. The asbestos is more readily prepared than paper. The tubes are easily made by a chemist and may be used any number of times. The small amount of cotton employed can be purified in the extractor. The cotton and the ignited asbestos are kept in bottles ready for use. This, I think, is less trouble than to purify by extraction the relatively large volume of paper necessary in the Adams method. With reference to purifying the paper P. Vieth

* Second Annual Report of the N. Y. Agricultural Experiment Station, p. 168; Report the Department of Health, of Brooklyn, 1885, p. 112; U. S. Department of Agriculture, Division of Chemistry, *Bulletin* No. 16, p. 76.

† London *Analyst*, 1885, p. 46.

says:* "It is indispensable that the paper used in the process should be most thoroughly exhausted from matter soluble in ether."

2d. Babcock's method I consider more accurate. In the use of asbestos I have not known of the question of accuracy being raised. This cannot be said with reference to the paper method. Unlike asbestos, paper is an artificial substance; while it consists principally of cellulose, it may contain various other organic substances. At the meeting of the Association of Official Agricultural Chemists, in August last, Dr. W. McMurtrie said that he had found in the paper used by him "a waxy, resinous substance, with difficulty soluble in ether."† In this case it is precisely those substances which are soluble with difficulty that are to be feared. Were they easily soluble they would be easily removed. In making the Adams method an official method the above association says: "It gives uniformly about 0.2 per cent. more fat in normal milk than the ordinary gravimetric methods."‡ Dr. Vieth made comparative determinations of fat in sixteen samples, using the plaster of paris and paper methods. He continues as follows (*loc. cit.*): "On comparing the results of the two methods it was found that in all and every case a higher percentage of fat was obtained by using the paper method, the plus ranging from .07 to .46, and averaging .271."

From this it would appear that there is a considerable uncertainty in the process, and that a maximum error of nearly one-half per cent. may result. From the complex and uncertain composition of paper it could not be expected that this discrepancy would be uniform.

3d. An advantage in the use of asbestos arises from the secure condition of the milk, after it is once introduced into the tube and during the process of drying. It may be handled in any manner, and nothing short of rupture of the tube will lead to loss; and

* London *Analyst*, 1887, p. 62.

† U. S. Department of Agriculture, Division of Chemistry, *Bulletin* No. 16, p. 30.

‡ U. S. Department of Agriculture, Division of Chemistry, *Bulletin* No. 16, p. 74.

further, as Dr. Babcock says, after once heating, the milk is sterilized and the process of drying and extracting may be completed at any subsequent time.

4th. The total solids may be determined on the ignited asbestos without the further trouble of previously drying to a constant weight.

Some recent experiments on the necessary amount of drying and extracting will be given in another paper.



THE UNITED STATES GALLON.

“The gallon is a vessel containing 58372.2 grains (8.3389 pounds avoirdupois) of the standard pound of distilled water, at the temperature of maximum density of water, the vessel being weighed in air in which the barometer is 30. inches at 62° F.” —(See report on “Weights and Measures,” by Secretary of the Treasury, Senate Doc., 1857.)

This definition being somewhat obscure, occasion was taken to write the Treasury Department, from which letter the following is quoted:

“Does this wording mean that the ‘gallon’ is a *volume* equal to that occupied by the above quantity of water under the conditions named; or does it intend a ‘gallon’ to be understood as meaning the quantity of liquid capable of being held by such standard *vessel* as above referred to, even after said vessel has been expanded by elevation of temperature? I take it that the latter view, although certainly stated, can hardly be intended.”

The reply to this came from the office of the “Coast Survey,” and read as follows:

“The standard gallon is a measure of capacity or volume. Its capacity was derived from standard weights, and in verifying and standardizing capacity measures it is the practice to determine the volumes by weighings. It is, therefore, to be understood that the capacity of a gallon is measured by the weight of a volume of distilled water at maximum density, whose weight in air at 62° F. Bar. 30. inches, equals 58372.2 grains.