NOTE ON THE VALENTA ACETIC ACID TEST.

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VALENTA'S test has given in our hands such general satisfactory results that we are somewhat surprised that it has not held a more important position than it appears to have done. The acetic acid turbidity temperature has the undeniable disadvantage of being one of those empirical methods of analysis which cannot be regarded with the same satisfaction as the many estimations which are so definite as to be expressed in the form of a chemical equation. We contend, however, that notwithstanding this fact, the value of this test is by no means small, and, further, that this disadvantage equally applies to the Reichert-Wollny, the Maumène and other important determinations.

Certain precautions have to be noted in order to obtain concordant observations, and we wish to call attention to the following points :

- (1) Strength of acetic acid.
- (2) Presence of water in the oil or fat under examination.
- (3) The temperature to which the oil or fat has been previously heated.
- (4) The method of stirring and observing the turbidity point.

1. We have paid considerable attention to the most suitable strength of acid to use, and after a somewhat lengthy trial have adopted an acid which contains 99.50 per cent of acetic acid.

In this connection we would like to call attention to the remarks made by Mr A. H. Allen in his "Commercial Organic Analysis," vol. ii., p. 26, in which he says that a slight variation in the strength of the acid employed is not of great importance. This is not in accordance with our experience, when working with an acid of the above-stated strength, and we believe that as the amount of water is increased the extreme sensitiveness of the turbidity test is diminished. On the other hand, there are certain reasons why an absolute acetic acid should not be used. It will be seen from this that acid of the strength specified by Valenta is more liable to give a little latitude in the exact strength of the acid to be employed, but that while such is the case, this advantage is entirely outweighed from the standpoint of sensitiveness.

2. The presence of moisture in the fat or oil under examination is probably one of the most fruitful sources of error, and we have found that in all cases it is advisable to filter the samples through a perfectly dry filter paper before making the test. It is well known to oil importers that the presence of minute quantities of water in the oil seriously affects the appearance of the oil as soon as the temperature falls, and that an oil may be quite brilliant and yet contain an estimable quantity of water. We believe that this fact was not quite recognised by Valenta, and also that it explains, or partly explains, the difference in figures obtained by Valenta in the same class of oil.

Olive-oil has shown the most serious variations in this respect, and it is the oil most likely to contain traces of water, owing to the methods of pressing and the variable condition of the fruit at the time it was pressed.

3. When an oil or fat is unduly heated, either at the time the test is being made or previously, the determination of the turbidity figure must not be relied upon. In all cases the sample must be dealt with as cautiously as it can be in this respect, and we would recommend that the temperature of 100°C. should never be exceeded in the preparation of an oil or fat for the test.

4. The method which is usually adopted consists in stirring together by means of a thermometer equal volumes of acetic acid and the sample in a test-tube after the solution is effected, removing the source of heat, and noting the temperature at which turbidity appears.

This is very well as a rough sorting test, and answers well enough in many cases; but we have now adopted the plan of conducting the test in a bath of water which has been raised to a suitable temperature; by these means we obviate an error which arises from the unequal cooling of the test-tube, and instead of a kind of tiny

waterspout appearing in the tube, the whole of the contents of the tube become turbid instead of doing so locally.

We would like to call attention to a comparatively perfect method of working the test, which, notwithstanding its extreme simplicity, is capable of giving results which may be regarded as absolutely concordant.

In some methods of analysis it is not expedient to arrive at very exact results owing to length of time necessary, but we hope it will be generally regarded by this society that a simple test like this, which enables a reliable, and even quantitative, result to be obtained in a few minutes, will allow of a little time being spent in order to obtain highly concordant figures.

Instead of using an ordinary test-tube, we use a short and somewhat thick test-tube of about $4'' \times \frac{1}{2}''$, into which a well-fitting stopper has been ground. We then weigh into the tube 2.75 grammes of the fat, and measure from a burette, or other suitable arrangement, 3.0 c.c. of the acetic acid. The tube is then stoppered, and placed in a beaker of warm water, increasing the heat until, after well shaking the tube, the contents become quite clear. The source of heat is then removed, and the test-tube so placed that it is in the centre of the beaker of heated water, and by means of a thermometer attached to the test-tube by a rubber band, the whole is allowed to rest until we note the change from brilliancy to turbidity. The change is very definite, and can be repeated over and over again with a maximum error of about 0.25 of a degree. If preferred, the thermometer may be used as a thermometer stopper to the test-tube, but it is not in our opinion necessary, as it is not always convenient to have a few dozens of thermometer-stoppered tubes, whereas simply stoppered tubes are more easily obtained and equally satisfactory.

It occurs to us that the foregoing remarks may appear to reflect to some extent upon the value of the Reichert-Wollny and other determinations. Such is very far from our intention. Food analysis is now no simple matter, and every care has to be exercised before one is in a position to state whether a food sample is genuine or not.

We realize that every determination that can throw light, however small, upon the analysis of food, and particularly of such commodities as butter and cheese, is most valuable. In this sense, and in this purely, we would suggest that the use of this method of analysis be taken up by those who have not already done so.

Finally, we do not wish to place the Valenta test in too high a position. There are still some oils which give strange and apparently inconsistent results.

The investigations into these irregularities are already under our consideration, and we believe that they are capable of simple explanation in most cases. It must not, however, be supposed that an oil may be purified by several methods, and that then each of such purified samples will give the same result. We repeat that this is a sensitive test, and as such it is quite inconsistent to expect that under such conditions as those cited we should not get a different result.

Its use as a valuable indication as to the nature of butter, margarine proper, and mixtures of these is now established.

We have placed on the blackboard just one instance of this—a sample of margarine, and of butter, and definite mixtures of the same.

The calculations are based upon the figures obtained from the butter and margarine proper as initial figures; but these figures are quite within the usual range, and these determinations were made without any important care, such as that specified in the improved method to which we have referred:

PRESENT.			FOUND.	
Mixture.	Butter.	Margarine.	Butter.	Margarine.
1	10	90	8.6	91.4
2	20	80	20.7	79.3
3	30	70	31.0	69.0
4	40	60	41.3	58.7
5	50	50	50.0	50.0
6	60	40	58.6	41.4
7	70	30	70.7	29.3
8	80	20	81.0	19.0
9	90	10	93.1	6.9

The following table contains some further figures taken upon a variety of pure oils, which we think may be of some interest. These results, however, must not be regarded as standards:

ACETIC ACID TURBIDITY TEST.

	°C.		°C.		°C.
Butter-fat :		Cottonseed ...	75.0	Japan Fish	19.0
(Number of samples		" ...	71.0	Herring ...	90.0
examined, 24)		" ...	85.0	Nigerseed ...	68.5
Highest ...	39.0	" ...	86.0	Sunflower ...	59.0
Lowest ...	29.0	" ...	89.0	" ...	62.5
Mean ...	36.0	" ...	88.0	Bottlenose ...	80.0
Margarine :		Cod ...	31.0	" ...	96.0
(Number of samples		" ...	29.0	Lard-oil ...	75.0
examined, 5)		" ...	26.5	" ...	76.0
Highest ...	97.0	" liver ...	76.0	" ...	75.0
Lowest ...	94.0	" " ...	72.0	Lard ...	98.0
Mean ...	95.5	" " ...	73.0	" ...	97.0
Olive (Bari) ...	91.0	Colza (German) ...	83.0	" ...	98.0
" (Smyrna) ...	91.0	Rape ...	63.0	" ...	97.0
" (Lucca) ...	90.0	" ...	77.0	Neat's-foot ...	72.0
" ...	91.0	" ...	77.0	Tea ...	78.0
" ...	89.0	" ...	78.0	Rosin ...	56.0
" ...	86.0	Peach-kernel ...	82.0	" ...	42.0
" ...	85.0	" ...	82.0	Jamba ...	above 100.0
" ...	90.0	Ground-nut ...	72.0	" ...	100.0
" ...	83.0	" ...	73.0	" ...	100.0
" ...	85.0	" (Arachis) ...	73.5	Cabbage ...	100.0
Almond ...	87.0	Linseed ...	46.0	Beef-stearin ...	100.0
" ...	84.0	" ...	52.0	Lard " ...	100.0
" ...	82.0	" ...	48.0	Castor not above	18.0
" ...	82.0	Seal ...	70.0	Woolgrease olein	
" ...	72.0	" ...	65.0	not above	18.0
Cottonseed ...	71.0	Japan Fish	47.5		