Salt - The mineral matter can be extracted from the curd and salt residue by extraction with water. Water is added to the residue, filtered, and the residue on the filter paper washed with water. The amount of chloride in the filtrate is estimated by titration with standard silver nitrate solution.

Butters containing less than 2% of salt are usually classed as fresh or mild cured: Those with more than 2%, but usually about 4%, are called salt butters. These may contain up to 9%.

Fat. - The percentage of fat is most conveniently obtained by substracting the sum of the percentage of water and of curd and salt from 100.

The Detection of Borates in Butter or Margarine. A small sample of butter is melted, and a few cc. of water made acid with HCl are added and mixed whilst warm. The water is allowed to separate, poured through a wet filter paper. The filtrate is tested with turmeric paper as given under milk.

Coburing Matters. - The commonest colouring matters of butter and margarine are annatto and butter yellow (dimethyl amido azobenzene).

Annatto or turmeric may be detected by extracting with dilute alkali and testing as for milk.

Butter yellow is extracted by shaking the warmed fat with an equal bulk of HCl, when a strong pink colour develops which disappears on cooling.

Butter fat is the fat of milk. It is composed almost entirely of triglycerides of the fatty acids. The characteristic constituent is the radicle of butyric acid (3-6%).

Fat Analysis.

Although the application of a few simple tests will often serve to differentiate between a pure butter and margarine, the detection of small amounts of margarine or lard in an unknown butter is often very difficult.

When melted in a water oven at about 50°C. the curd of butter usually settles down with the water, leaving an upper layer of almost clear fat; with margarine the upper fat layer remains turbid until after filtration.

The Valenta Test. The glycerides of saturated fatty acids are usually soluble in warm pure acetic acid but their solubilities vary and so they tend to come out of solution at different temperatures. The temperature at which a solution of a fat in acetic acid begins to deposit solid glycerides varies with the constitution of the fat. Butter itself shows a turbidity within reasonably close limits (34° - 44°) under strict conditions of experiment, and pure margarines show similar close limits but at a much high temperature 90° - 98° , but always above 80°).

A method for carrying out the test is a follows:

A long thin hard glass tube is marked so that 3 cc. and 6 cc. quantities can be accurately measured into it.

About 5 g. of butter are placed in a test tube and stood in a beaker of water at about 50° - 60°. When the water has settled small pellets of dry filter paper are added and allowed to soak up the water. A small wad of cotton wool is now pushed through the fat. The supernatant water free fat after heating to remove water is now poured into the long thin tube (placed in the bath) until just 3 cc. have been added.

3 cc. of pure acetic acid are now added to the 6 cc. mark.

A thermometer with a very small bulb is fixed in the tube, with its bulb at the 3 cc. mark.

The fat is dissolved in the acetic acid by shaking in water at about 50°C.

When dissolved the tube is withdrawn and the contents allowed to cool in the air, shaking gently and holding the tube in a good light.

Immediately the faintest turbidity appears the temperature is read.

The tube is then slightly warmed and a fresh reading obtained.

For margarine the mixture will have to be heated to a higher temperature, about 100°; before the fat goes into solution.

Important

Detection of Adulteration of Butter Fat.

The fats most commonly used to adulterate butter are lard, oleo products and oils of the coconut group. If butter is entirely substituted by a margarine the detection is easy but if 10% addition of some of these fats is made then the detection is not very easy and several tests must be made. If, however, the fats are examined in this way even the most skillful adulteration can usually be detected. The most reliable examination is made by the Reichert-Meissl, Polenske, Kirschner method. Before detailing this method it is advisable to consider the constitution of the fats.

Fats. A fat is actually a compound of one or more fatty acids with glycerol (glycerine). Glycerol and a trihydric alcohol, i.e. it is an organic compound containing 3(OH) groups, and each of these groups may be replaced by a fatty acid. Glycerol may therefore combine with three molecules of the same fatty acid or with one molecule of each of three distinct fatty acids. Thus if we represent glycerol as $G(OH)_3$ then we may have fats constituted as follows

$$G(O.CO.R)_3, \qquad G\begin{cases}(O.CO.R_1)_2\\(O.CO.R_2)\end{cases} \qquad \text{or} \qquad G\begin{cases}(O.CO.R_1)\\(O.CO.R_2)\\(O.CO.R_3)\end{cases}$$

where R_1, R_2 and R_3 represent the radicles of fatty acids. If then we could take a fat and split it up into glycerol and its fatty acids then an examination of the amounts of the different fatty acids obtained might give us an idea of the origin of the fat. This is the course followed in fat analysis. The fat is split up or hydrolysed by heating with caustic soda, and the fatty acids are then examined. Butter fat is unlike most other fats in containing a large proportion of glycerides of the lower fatty acids such as butyric acid and hence the determination of the amount of butyric acid is important.

In the Reichert-Meissl, Polenske, Kirschner method of analysis three determinations are made, namely of the Reichert-Meissl value, the Polenske value and the Kirschner value.

The Reichert-Meissl value is a determination, under certain standard conditions, of the amount of volatile, water soluble, fatty acids obtained from 5 g. of butter fat. Butter aver 27 Margarine 8.0 or 5.2 Coconut Palm Kernel

The Polenske value represents the amount of volatile, water insoluble fatty acids obtained under similar standard conditions, whilst

Butter average 2.4 Margarine 16.5 or 9.9

The Kirschner value is a measure of the proportions of butyric acid and the higher volatile, water soluble fatty acids present in the fat.

Butter aver 23.5 Margarine 1.8. 1.0

Detection of adulteration of Butter

Important

Briefly the method may be summarised as follows:

(a) The butter fat is dried and then hydrolysed with caustic soda. The fat is split up into glycerol and sodium salts of the fatty acids, thus

$$G(O.CO.R)_3 + 3NaOH = G(OH)_3 + \underset{\text{(sodium salts)}}{3R.COONa}$$

(b) This mixture is then acidified with H_2SO_4 to set free the fatty acids from their salts

$$2R.COONa + H_2SO_4 = 2R.COOH + Na_2SO_4$$

Some of these free fatty acids are volatile and some are not.

(c) The mixture is now boiled and the volatile fatty acids distilled over, the distillate containing all the volatile acids, including both water soluble and water insoluble acids.

(d) The distillate is filtered through dry filter paper so that the insoluble acids are kept back.

(e) The soluble acids in the filtrate are titrated, c n/10 NaOH giving the Reichert-Meissl value. 27

(f) The water insoluble volatile acids are dissolved in neutral alcohol and titrated, giving the Polenske value. 2.4

(g) The neutralised water soluble acids of (e) are treated with Ag_2SO_4 which precipitates the higher boiling point acids as their silver salts. The mixture is filtered.

(h) The mixture is filtered, the soluble silver salt of butyric acid and the other low boiling point acids passing into the filtrate.

(i) The filtrate is acidified and the freed butyric acid, etc. distilled and estimated by titration. From the Reichert-Meissl value can be calculated the Kirschner value.

X

Details of the Reichert-Meissl, Polenske, Kirschner Method.

In actual practice it is essential that the whole of this method be standardised and that the apparatus used be of standard measurements throughout. The following are the experimental details of the method.

(1) Place 5 g. of a melted and filtered sample of butter or other fat into the standard 300 cc. flask, add 2 cc. of the aqueous NaOH (1:1) free from CO_2 and 20 g. of glycerine.

(2) Heat the flask over a bunsen burner with continual shaking until the fat is completely saponified, as will be indicated by a sudden clearing of the soap.

(3) Cool to below 100° and add 100 cc. of hot water, which has been kept boiling for at least 10 minutes.

(4) Add 0.1 g. of finely powdered pumice and 40 cc. of dilute H_2SO_4 of such strength that 35 cc. neutralise 2 cc. of the soda solution, (about 20 cc. strong acid to the litre).

(5) Connect the flask with the standard distillation apparatus.

(6) Heat over a small flame until the insoluble fatty acids are completely melted.

(7) Increase the flame so that distillation of the volatile acids takes place at the rate of approximately 110 cc. in 20 mins.

(8) After collecting 110 cc. [see (11)] cool the distillate in water at 15°C. for 15 minutes and filter. Keep the flask and filter paper.

(9) Titrate 100 cc. of the filtrate with N/10 alkali (baryta if the Kirschner value is to be determined) using phenol phthalein as indicator. (Keep the neutralised liquid for the Kirschner value determination).

(10) The Reichert-Meissl value = no. of cc. N/10 alkali required x $\frac{11}{10}$

(11) A small beaker is placed under the condenser when the distillation is completed. [(8) above].

(12) Wash the condenser, beaker and the 110 cc. receiving flask with small quantities, e.g. 3 lots of 6 cc. of water and filter these washings through the filter kept from (8). Reject the washings.

(13) Now wash the condenser, etc. with 3 lots of 10 cc. quantities of warm neutral alcohol and pass through the filter The alcohol dissolves the insoluble acids.

(14) Titrate the mixed alcohol washings with N/10 alkali, using phenol phthalein as indicator. The number of cc. of N/10 alkali required is the Polenske value.

(15) To the neutralised 100 cc. of distillate containing the water soluble, volatile fatty acids from (9) add 0.5 g. Ag_2SO_4.

(16) Leave the mixture standing for at least an hour, with occasional shaking. Filter.

(17) To 100 cc. of the filtrate add 35 cc. of water, 10 cc. of dilute H_2SO_4 and a small length of aluminium wire (10 cm.).

(18) Distil in the standard apparatus at the rate of 110 cc. in 20 mins.

(19) Titrate 100 cc. of the distillate with 0.1N. alkali.

(2)) After deduction of any "blank" value, the Kirschner value is calculated from the equation.

$$K = \frac{121X(100 + y)}{10,000}$$

where X = titration (less blank)

and y = no. of cc. of N/10 alkali added to neutralise the original 100 cc. of Reichert-Meissi distillate.

The following figures give values for butter and margarine.

Value	Butter	Margarine.	
		Coconut oil.	Palm Kernal oil.
Reichert Meissl	24.33 Average 27	8.0	5.2
Polenske	1.5-3.7 " 2.4	16.5	9.9
Kirschner	20-26 " 23.5	1.8	1.0

Margarine may be prepared from animal or vegetable fats or oils. The most common bases are coconut and palm kernal oils. Butter fat is often added to improve the flavour but the Sale of Butter Regulations, 1907, limits the addition to 10%. The methods of examination are the same as those given for butter.

Cheese may be made from the milk of any animal, but the great majority in commerce are made from that of the cow. Rennet (41°) is added to the milk, which causes the casein to be split up into 2 compounds, one of which is soluble, the other in the presence of Ca phosphate being insoluble; this latter carries down with it most of the milk fat as well as some of the milk sugar. The thin whey is allowed to run off and the precipitated "curds" submitted to pressure. Cheeses may be divided into 2 classes, the soft and the hard. The former are made by precipitating with rennet at a low temperature and using little pressure; they have mostly an alkaline reaction. The hard cheeses are subjected to a higher temperature and stronger pressure, and have, when first made, an acid reaction. Soft cheeses may be made from whole milk, partly skimmed milk or from milk and cream. True cream cheeses contain upwards of 75% fat when calculated onthe dry basis, whole milk cheeses similarly calculated show 45-50% or more fat.

Lard is the fat of the pig but strictly speaking it should be the solid fat from around the kidneys and from the peritoneum.

Important

$[R.COO]_3$ G. — Fat = an ester of an alcohol, glycerol and a fatty acid.

Saponification NaOH ↓

3 R.COONa + $G(OH)_3$ glycerol.

↓ H_2SO_4

R.COOH + R COOH — Free acids, volatile and nonvolatile.

↓ distil

R.COOH — Volatile acids, soluble and insoluble in water.

↓ filter. Dissolve insol acids in alcohol → Polenske (a measure of water insol. vol. acids.)

R. COOH. — Water sol. vol. acids.

↓ Filtrate.

R.CooNa — (Reichert Meissl).

↓ Ag_2SO_4 and filter.

R.COOAg (Ag salt of Butyric acid) → H_2SO_4 and distil → R.COOH. Butyric acid. → HaOH and calc. → Kirschner.

~~Saponification~~ Iodine number is the amount of milligrams of Iodine taken up by one gram of fat.

Saponification number or equivalent is the number of milligrams of Na— or K— OH used to completely saponify one gram of fat

10cc = 2.1 cc.

30 cc = $\frac{30}{10} \times 2.1$

= 6.3 cc N/10 NaOH

$\frac{6.3 \times 5 \cdot 8 \cdot 5}{1000 \times 4.57}$

.8% ash

Wt of dish = 45.31 gm.

Wt of dish + fat extract = 45.51

∴ fat extract = 45.31

.2 gm.