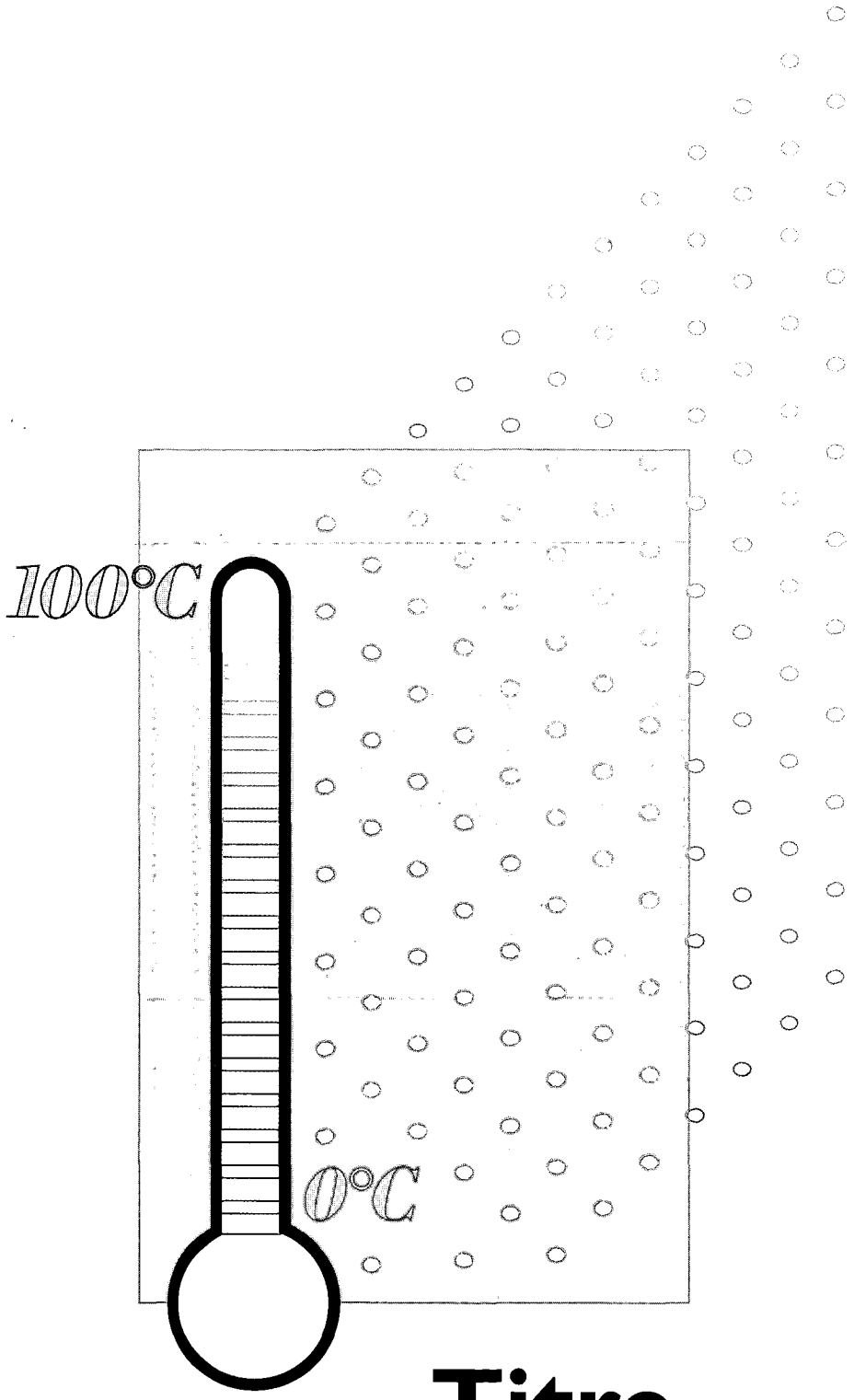


# Titre

1998

Meat Research Corporation

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**Titre is a required specification in sales contracts for most export tallows. It is a measure of the average solidification temperature of the fatty acids derived from the fat sample. Titre is fixed by the fatty acid composition of the fat and so gas chromatography, which gives a complete profile of fatty acids in a fat, can replace the need to test for titre. Titre is also an indication of the relative hardness of fats. For example tallows produced from tissues from different parts of an animal or from different species have different titres and differ in hardness. The type of environment in which livestock or plants are grown may influence the titre of fats produced by the animals or plants.**

Results of titre tests are distinctive and reproducible and can be used to confirm the type of fat stated in a sales contract, or as a check on contamination with other fatty materials. Terms of sale such as the USA trading rules define animal fats with titres above 40°C as tallows, and those below are classed as greases. Soapmakers rely on titre specifications to produce consistent quality soaps.

## Application

Determination of titre is important in tallows and other fats used for soap and oleochemical manufacture. It as also applied to most sales contracts of bulk quantities of tallow sold as a commodity. The test method described below is optimised to specially suit the following types of tallows and titre ranges:

- beef tallow with titre 41°C to 45°C,
- mixed beef and mutton tallow with titre 42°C - 44°C
- mutton tallow with titre 44°C - 48°C
- pig tallow with titre 36°C - 40°C
- poultry oil with titre 30°C - 38°C

The test is applied to almost all international or domestic purchase contracts when proof of purity or conformity with a particular type of tallow is specified. It is used as a means of confirming the species of origin of a tallow.

The titre of fats is influenced by contamination with foreign fats and the process of fractionation. In fractionation the higher melting point fat fraction solidifies and separates from the oilier lower melting point fats. The separate fractions will have different fatty acid compositions and different titres. If tallows are partly solidified or if they have been allowed to cool slowly and become a seedy mixture of fat crystals in liquid fat, they cannot be satisfactorily sampled for titre testing. It is best to heat the bulk tallow to at least 10°C above the apparent melting point and then mix it well to ensure it is homogeneous before drawing off a sample.

## Outline of method

Fats are mixed esters of glycerol combined with many types of fatty acids. In the titre test, fats are first saponified in hot glycerol by the action of strong potassium hydroxide (caustic alkali). This produces liquid potassium soaps of the fatty acids. The fatty acids are released as free fatty acids from the soaps by acidulation with dilute sulphuric acid. The free fatty acids are insoluble in the water/glycerol mixture and can be separated and purified. The purified molten fatty acids are cooled slowly in a controlled manner so that the temperature can be observed at the point when the energy released by the solidifying fatty acids causes a small rise in the temperature of the mixture. The maximum point of the temperature increase is called the titre.

### Precautions

**Use adequate protective clothing and great care when handling strong acids and alkalis. Accidents with acids and alkalis can cause blindness and severe burns.**

Specially made mercury in glass titre thermometers must be used. These thermometers are finely graduated and cover a range from 0 - 69°C. They must be handled carefully and calibrated annually.

The ambient temperature in the laboratory must be normal i.e. controlled within the range 20 - 28°C to produce the required differential with the titre of tallows.

Microwave ovens are useful to preheat fat and glycerol solutions and to boil aqueous solutions, usually on lower power settings. However fatty acids do not absorb microwave energy and cannot be heated in a microwave oven. The initial

reaction between the glycerol caustic solution and tallow must be carried out for at least 30 minutes while boiling the reaction mixture on a hot plate. A microwave oven may be used to maintain temperature at 125°C for the remainder of the reaction time.

It is important that after addition of the acid, the mixture is well stirred, without splashing, so that when the fatty acids are fully melted, a clear upper layer of pure fatty acids forms.

Hot liquids used in the method can cause severe burns and must be handled with appropriate eye and hand protection. All activities producing an evolution of fumes must be conducted in a fume cabinet.

## Alternative methods

The AOCS Cc 12-59 method should be consulted for the official USA trading rules.

Also refer to ISO 935:1988, IUPAC 2.121, BSI (U.K.) and ASTM.

## Equipment and Suppliers

All equipment and basic reagents can be obtained from major laboratory suppliers.

## Method

### Equipment

- Hotplate, magnetically stirred and thermostatically controlled in range 50 - 150°C.
- 800ml heavy-duty heat-resistant glass beaker.
- 250 and 150ml glass beakers.
- 250ml conical flask fitted with 10 - 20mm thickness of cotton wool on the bottom to support the titre test tube. The conical flask with titre tube fitted is shown in Figure 1 .
- Titre test tube with diameter 20mm and length 130 -150mm. The tube should be fitted with a cork or rubber stopper with holes drilled for the thermometer and a stirring device as shown in Figure. 1. Sit the tube on the layer of cotton wool on the bottom of the flask.
- The stirring device is made from steel wire, about 2mm thick with a lower loop approximately 15 mm in diameter to freely encircle the thermometer.
- Stirring thermometer, 10 - 150°C
- Titre thermometer, mercury in glass, calibrated annually. Use AOCS H 6-40-68°C or ASTM 36°C - 2 - 68°C in 0.2°C divisions and marked for 45mm immersion.
- Glass filter funnel, 100mm diameter.
- Filter paper, 180mm diameter, Whatman 41 grade or similar.

### Reagents

**CAUTION: Use vinyl gloves and goggles during these preparations.**

- Glycerol caustic solution:

  Slowly add 100 grams of potassium hydroxide (80%) with stirring into 500 grams of A.R. glycerine at 120°C. Do not heat above 145°C.

- Dilute sulphuric acid, 30% w/w:

  Prepare 160ml of concentrated sulphuric acid (specific gravity 1.84) in a 250ml beaker and gradually pour it, with stirring and caution, into 700ml of cold distilled water.

- Anhydrous sodium sulphate AR.
- Methyl orange indicator solution, 1% in water

### Procedure

1. Weigh about 38 - 40g of well mixed tallow into a 150ml beaker and heat and hold at about 65°C.
2. Weigh 110g of glycerol caustic solution into a 800ml beaker, heat to 120°C and add the pre-weighed tallow sample.
3. Mix thoroughly using a sturdy glass stirring rod for 1 minute.
4. Heat the mixture to 140 - 150°C maximum using an electric hot plate, and stirring with a large spin bar for approximately 1 hour. The solution should be completely clear after saponification is completed. Signs of saponification are small soap


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bubbles forming on the surface and that the liquid becomes thinner after a thickening phase.

5. Cool to 90-95°C, add 300ml of hot water (approx. 70°C) and stir for one minute.

6. With constant stirring, add 50ml of dilute sulphuric acid. Test a sample of the solution to ensure that it is acidic: withdraw a little of the solution with a Pasteur pipette and add one drop of methyl orange. The methyl orange will turn red when the solution is acidic. Continue stirring at moderate speed (avoid spattering) until the fatty acids form a completely CLEAR upper layer when stirring slowly.

7. Stand the mixture in a cool place (e.g. refrigerator) until the top layer of fatty acids solidifies. Remove the solid disk of fatty acids and wash it under cold water. Put the disk in a beaker with a little water and heat until the fatty acids melt. Repeat the step of cooling the washed fatty acids and recovering the solid disk of fatty acids.

8. Dry the disc of fatty acids on a paper towel or filter paper. Place fatty acids into a clean 150ml. beaker and heat to 80°C.

9. Add 3 - 4g of anhydrous sodium sulphate and mix. Filter the mixture through a Whatman 41 fluted filter paper and collect the filtrate. Dry the filtered fatty acids by heating to 130°C maximum then transfer into a 20mm diameter glass test tube to a level of 40mm.

10. When the fatty acids have cooled to about 10°C above the expected titre, place the titre thermometer into the fatty acids so that the bulb sits in the centre and at mid depth, with stirrer wire at the bottom. Place the titre tube in the apparatus as shown in Figure 1. and commence stirring using a steady up and down movement of about 30mm at 90 -100 strokes per minute until a slight cloudiness is observed. Immediately withdraw the stirrer from the liquid and record the temperature at 30 seconds intervals. DO NOT DISTURB THE APPARATUS DURING THIS PERIOD. Ensure the eye is level with the mercury surface when reading the thermometer.

11. The titre is the maximum temperature reached in this phase before falling again (preferred result) or it is the temperature that remains constant over 30 seconds. Repeated determinations on the same preparation should agree within ±0.2°C.

***Notes:***

If there is an indistinct increase in temperature at the titre point, or if the result is lower than expected, the sample could be wet or impure. Reheat the test tube of fatty acids in an air oven or in a beaker of hot water and repeat steps 9 and 10.

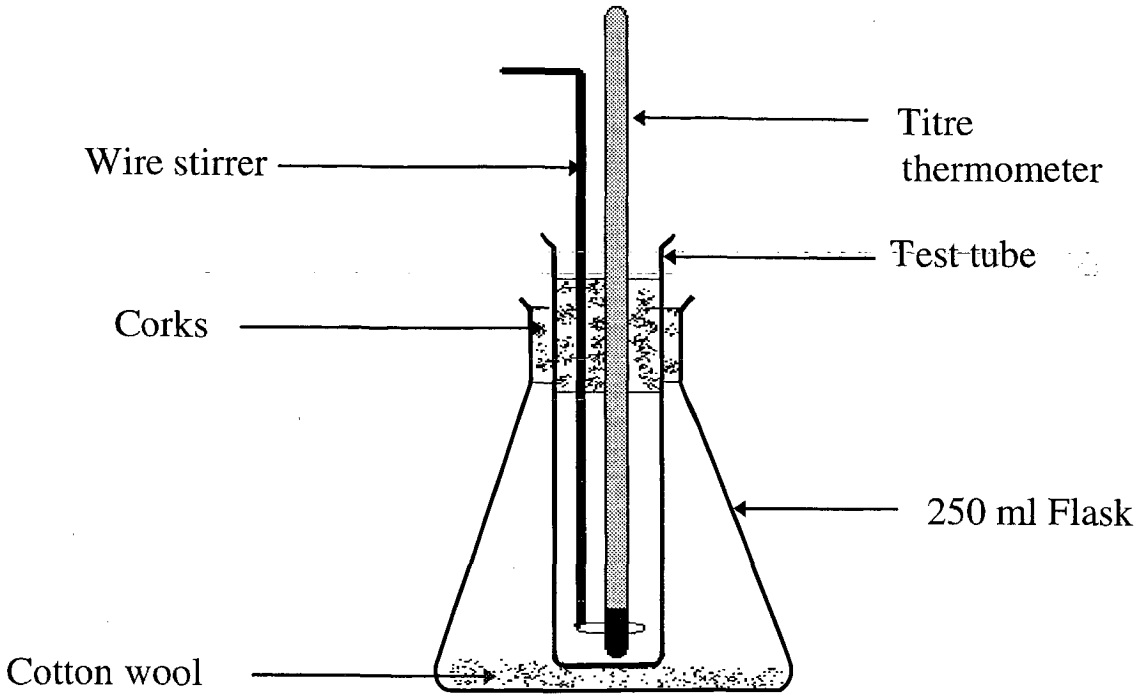
Otherwise repeat the whole preparation ensuring a longer boiling time to complete the saponification reaction, and check reagent strengths.

Complete saponification is difficult to detect but it can be indicated by a clear reaction medium. Also there should be no steam rising from the beaker as tested by placing a watch glass over the beaker. The reaction temperature maximum of 145°C must be achieved.

## References

'Official Methods and Recommended Practices of the American Oil Chemists Society' (1993). Edited by D. Firestone. AOAC Press, Illinios.

FIGURE 1 Titre apparatus




## Additional information

Additional help and advice are available from Food Science Australia, Meat Industry Services Section:

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