Australian Standard®

Methods of chemical and physical testing for the dairying industry

Method 2.7: Liquid milks—Determination of calcium

PREFACE

This Standard was prepared by the Standards Australia Committee FT-024, Food Products and Subcommittee FT-024-05, Dairy Products to supersede AS 2300.2.7—1988.

After a periodic review, the Committee recommended a new edition. This edition confirms the method without technical changes, but updates the referenced documents and reflects the current editorial style and includes a clause on uncertainty of measurement.

AS 2300 comprises a series of methods and related Standards for chemical and physical testing of milk and dairy products, including the preparation of samples for testing.

Standards in the AS 2300 series are divided into categories according to type of product to be tested, as follows:

AS	
2300.1	General methods and principles
2300.2	Liquid milks
2300.4	Dried milk and dried milk products
2300.5	Condensed milk
2300.6	Cheese
2300.7	Butter
2300.8	Anhydrous milk fat
2300.9	Analysis of ice-cream and frozen milk products
2300.10	Caseins, caseinates and coprecipitates
2300.11	Cultured milk products

FOREWORD

This method is based on classical procedures, but it should be recognized that other procedures using more advanced instrumentation are available, for example atomic absorption and inductively coupled plasma spectrophotometry.

METHOD

1 SCOPE

This Standard sets out a routine method for the determination of the calcium content of milk and of milk reconstituted from evaporated, condensed or dried milk.

2 APPLICATION

The method is applicable to raw milk, pasteurized milk, homogenized milk, reconstituted milk, skim or low fat milk, UHT milk, sterilized milk, and cream.

3 REFERENCED DOCUMENT

The following document is referred to in this Standard.

AS/NZS
2243 Safety in laboratories
2243.2 Part 2: Chemical aspects

4 PRINCIPLE

The protein in the sample is precipitated with trichloracetic acid. The calcium contained in the filtrate is precipitated as calcium oxalate which is separated by centrifuging, dissolved in sulphuric acid and the oxalate solution titrated with potassium permanganate.

5 REAGENTS

Use only reagents of recognized analytical reagent grade and freshly distilled water or water of equivalent purity. The following reagents are required:

(a) Acetic acid solution (20 percent V/V)—dilute 20 mL of glacial acetic acid (φ20 1050 kg/m3) to 100 mL.

(b) Ammonia solution strong—mix equal volumes of ammonia (φ20 approximately 900 kg/m3) and water.

(c) Ammonia solution, weak—dilute 2 mL of ammonia (φ20 approximately 900 kg/m3) to 100 mL with water.

(d) Ammonium oxalate solution—saturated solution at room temperature.

(e) Methyl red solution—0.5 g/L solution in 95 percent ethanol.

(f) Potassium permanganate solution (0.004 mol/L) (prepared, allowed to stand overnight and filtered)—standardized under acidic conditions.

(g) Sulphuric acid solution (200 mL/L))—slowly add with stirring 200 mL of sulphuric acid (φ20 1840 kg/m3) to 500 mL of water. Cool and dilute to 1 L with distilled water.

CAUTION: DO NOT ADD WATER TO ACID.

(h) Trichloracetic acid solution, weak—120 g/L solution.

(i) Trichloracetic acid solution, strong—200 g/L solution.

WARNING: THE USE OF THIS STANDARD MAY INVOLVE THE USE OF HAZARDOUS MATERIALS, OPERATIONS AND EQUIPMENT. THIS STANDARD DOES NOT PURPORT TO ADDRESS ALL THE SAFETY RISKS ASSOCIATED WITH ITS USE. IT IS THE RESPONSIBILITY OF THE USER OF THIS STANDARD TO ESTABLISH APPROPRIATE SAFETY AND HEALTHY PRACTICES AND DETERMINE THE APPLICABILITY OF LOCAL REGULATORY LIMITATIONS PRIOR TO USE. SEE AS/NZS 2243.2 FOR MORE DETAILS REGARDING LABORATORY SAFETY.

6 APPARATUS

The following apparatus is required:

(a) Ashless filter paper for slow filtration (small pore size).

(b) Burette graduated in 0.02 mL.

(c) Centrifuge capable of producing a force approximately 1400 times that of gravity.

(d) Cylindrical centrifuge tubes—plastic or glass. Tube capacity should be approximately 30–50 mL and be marked at 20 mL.

(e) Suction device with tube—to obtain the supernatant liquid.

7 SAMPLING AND SAMPLE PREPARATION

7.1 Sampling

Ensure that the test sample is typical of the bulk lot.

7.2 Sample preparation

Warm the sample to 35 ±5°C and mix thoroughly but gently by repeated inversion of the container, so that any cream layer is uniformly dispersed without churning the fat. After mixing, cool the sample to 22 ±3°C. Invert the container three or four times immediately before taking a test portion for the determination. Discard the sample if it cannot be mixed satisfactorily.

8 PROCEDURE

8.1 Blank test

At the same time as the determination of the calcium content of the sample, perform a blank determination on 20 mL of water using the method described below, but omitting the test sample.

8.2 Precipitation of protein from sample

The procedure shall be as follows:

(a) Weigh, to the nearest 0.01 g, approximately 20 g of the milk sample into a volumetric flask of 50 mL capacity. Record the mass of the test portion (m).

(b) Gradually add strong trichloracetic acid solution, shaking the flask during the addition, and dilute to the mark with this reagent. Stopper the flask and shake vigorously for a few seconds.

(c) Allow the flask to stand for 30 min then filter through an ashless filter paper to obtain a clear filtrate.

8.3 Precipitation and separation of calcium oxalate

The procedure shall be as follows:

(a) Pipette 5.0 mL of the clear filtrate from Clause 8.2(c) into a centrifuge tube, then add about 5 mL of the weak trichloracetic acid solution, 2 mL of the saturated ammonium oxalate solution, two drops of the methyl red solution and 2 mL of the acetic acid solution.

