Australian Standard®

Methods of chemical and physical testing for the dairying industry

Method 2.8: Liquid milks—Determination of chloride

PREFACE

This Standard was prepared by the Standards Australia Committee FT-024, Food Products and Subcommittee FT-024-05, Dairy Products, to supersede AS 2300.2.8—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, ion chromatography. A silver ion electrode may be used in place of the indicator solution.

METHOD

1 SCOPE

This Standard sets out a method for the determination of the chloride content of liquid milks. The method is applicable to raw milk, pasteurized milk, homogenized milk, reconstituted milk, skim or low fat milk, modified milk, flavoured milk, UHT milk and sterilized milk.

2 REFERENCED DOCUMENT

The following document is referred to in this Standard.

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

3 PRINCIPLE

A known amount of silver nitrate is added to the milk sample and, after digestion with nitric acid, the excess silver nitrate is back-titrated with potassium thiocyanate solution using an indicator or silver ion electrode.

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.

4 REAGENTS

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

(a) *Indicator solution*—prepare a saturated solution of ammonium ferric sulfate dodecahydrate ($NH_4Fe(SO_4)_2.12H_2O$) in water. Add sufficient cold colourless 5 mol/L nitric acid to bleach the brown colour.

(b) *Concentrated nitric acid*—(approximately 1420 kg/m^3).

(c) *Silver nitrate solution* (0.05 mol/L)—store in a brown bottle away from light. Standardize against anhydrous sodium chloride.

(d) *Potassium thiocyanate solution*—standardized to 0.05 mol/L as follows:

Pipette 10.0 mL of the silver nitrate solution into a 250 mL conical flask and add 30 mL of water, 1 mL of concentrated nitric acid and 1 mL of indicator solution. Titrate with the potassium thiocyanate solution until a faint reddish-brown colour persists on shaking and calculate its strength in mol/L KCNS.

5 APPARATUS

The following apparatus is required:

(a) *Calibrated burette or other device*—capable of reading to least 0.02 mL.

(b) *Silver ion electrode*—optional

6 SAMPLING AND SAMPLE PREPARATION

6.1 Sampling

Ensure the sample taken for analysis is typical or representative of the bulk lot.

6.2 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.

7 PROCEDURE

7.1 Blank titration

At the same time as the determination of the chloride content of the sample, perform a blank determination on 10 mL of water using the procedure described below, but omitting the test sample.

7.2 Analytical method

The procedure shall be as follows:

(a) Weigh to the nearest milligram, approximately 10 g of the milk sample into a 250 mL conical flask. Pipette 10 mL of the 0.05 mol/L silver nitrate solution into the flask, add approximately 10 mL of concentrated nitric acid (approximately 1420 kg/m^3) and mix.

(b) Add glass beads (anti-bumping granules) and gently boil the contents in a fume cupboard. Allow to simmer with occasional shaking until the precipitate of silver chloride is granular and the liquid is pale yellow in colour and clear except for fat globules. Cool rapidly to approximately room temperature whilst minimizing exposure to light.

(c) If indicator solution is used: without undue delay add approximately 60 mL of water and add approximately 1 mL of indicator solution. Titrate the residual silver nitrate with the standardized potassium thiocyanate solution from the burette, until a faint reddish-brown colour persists.

If silver ion electrode is used: without undue delay, add approximately 60 mL of water whilst shaking, titrate the residual silver nitrate with the standardized potassium thiocyanate solution from the burette until no silver ions are present.

(d) Record the volume of potassium thiocyanate solution added.

8 CALCULATION

From the difference between the sample and blank titrations, calculate the percent *m/m* of chloride present in the sample from the following relationship:

1 mL of 0.05 mol/L KCNS = 1.773 mg of chloride ion

$$\text{Percent } m/m \text{ chloride ion} = \frac{(B - T) \times C \times 3.546}{m}$$