

Australian Standard®

Method of chemical and physical testing for the dairy industry

Method 1.6: General methods and principles— Determination of pH

PREFACE

This Standard was prepared by the Standards Australia Committee FT-024, Food Products and Subcommittee FT-024-05, Dairy Products (Constituted) to supersede AS 2300.1.6—1989.

There is no ISO Standard other than ISO 5546 which covers only caseins and caseinates.

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

The term ‘normative’ has been used in this Standard to define the application of the appendix to which it applies. A ‘normative’ appendix is an integral part of a Standard.

METHOD

1 SCOPE

This Standard sets out a method for determining the pH value of dairy products.

2 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS

1166 Milk and milk products—Guidance in sampling

AS/NZS

2243 Safety in laboratories

2243.2 Part 2: Chemical aspects

BS

2586 Glass and reference electrodes for measurement of pH

3145 Laboratory pH meters

3 PRINCIPLE

The pH is measured with a glass electrode, a reference electrode and a pH meter calibrated with standard buffer solutions of known pH.

4 REAGENTS

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.

4.1 Phthalate buffer solution pH 4.0

Accurate to 0.01 pH units (see Appendix A).

4.2 Phosphate buffer solution about pH 7

Accurate to 0.01 pH units (see Appendix A).

NOTES:

- 1 The exact pH of the buffers at the proposed temperature of measurement must be known to two decimal places.
- 2 For routine use, buffer solutions of satisfactory accuracy are commercially available. In work where accuracy is vital, the buffer solutions should be checked against buffer solutions made up in accordance with Appendix A.

4.3 Water—distilled water or water of equivalent purity, free from carbon dioxide.

5 APPARATUS

5.1 pH meter

Accurate to 0.01 pH units, requiring two buffer solutions for calibration, and with facility for temperature compensation. The instrument shall be capable also of displaying in millivolts or displaying the percentage slope.

NOTES:

- 1 pH meter testers for checking the performance of pH meters are commercially available.
- 2 For further information on pH meters see BS 3145.

5.2 Electrodes

pH electrode suitable for measuring 0.01 pH units. Essential information on performance and maintenance of electrodes is given in Appendix B.

NOTE: Suitable electrodes are commercially available.

6 SAMPLES

6.1 Laboratory sample

It is assumed that the laboratory sample has been obtained, transported to the laboratory and stored in accordance with the requirements of AS 1166.

6.2 Preparation of test samples

6.2.1 Liquid samples

Mix the sample well. The pH may then be determined directly.

6.2.2 Solid samples

The pH shall be determined directly if practicable. If necessary, solids may have to be grated and mixed with an equal mass (unless otherwise specified) of distilled water to provide a uniform dispersion. In the latter case, allow the slurry to stand for at least 5 min.

6.2.3 Butter serum

Melt, at a temperature not exceeding 50°C, sufficient butter to provide a suitable amount of serum, ensuring that the butter is not exposed to this temperature for longer than 15 min. Separate the serum for pH measurement.

NOTE: The serum can be separated by mixing the melted butter, transferring to a stoppered centrifuge tube and centrifuging in an inverted position until the two phases separate. Maintain the tube in the inverted position and quickly chill until the fat solidifies. Carefully remove the stopper from the tube, and allow the butter serum to run into a suitable vessel for measuring the pH.

6.2.4 Dried milk products and caseinates

Dissolve and disperse 10 g of nonfat dried milk product, dry buttermilk or caseinate, or 13 g of dry whole milk, in 100 mL of water. Use this solution for the pH determination.

6.2.5 Caseins

If necessary, grind the casein sample to provide a particle size which will pass through a 500 µm sieve. Where grinding is necessary, the grinder used shall be such that undue heat is not developed. Do not use a hammer mill.

Weigh 5 ± 0.1 g of prepared casein into a 100 mL stoppered vessel. Add 30 mL of water, shake well and allow to stand for 20 min. Use the supernatant liquid for the pH determination.

7 PROCEDURE

The procedure shall be as follows:

- (a) Bring the calibrating buffer solutions and the test samples to the same temperature, which shall be between 20°C and 26°C (preferably $25 \pm 0.5^\circ\text{C}$).
- (b) Adjust the temperature compensator of the pH meter to the temperature of the buffer solutions and samples, and calibrate the pH meter using the phthalate and phosphate buffer solutions according to the instructions of the manufacturer of the meter.
- (c) Insert the electrodes in the prepared sample and record the pH to the nearest 0.01 pH unit when the reading has been steady for at least 15 s.

8 ACCURACY AND UNCERTAINTY MEASUREMENT

Estimate the uncertainty of test measurements based on the performance characteristics of the test method (trueness and precision), verification data, quality control data, previous experience or other relevant information.

The appropriate degree of rigour applied to estimation of uncertainty depends on the purpose of the test and customer (end-user) requirements.

NOTES:

- 1 An estimate of uncertainty is required when it is relevant to the validity or application of the test result or when requested by a customer.
- 2 Unless otherwise required, the measurement value should be stated together with the expanded uncertainty, calculated using a coverage factor of 2, to give a level of confidence of approximately 95%.
- 3 The measurement value and its uncertainty should both be reported in the same units, the measurement value rounded to be consistent with its uncertainty.
- 4 The form of reporting the result must not give a wrong impression of the uncertainty.

9 REPORT

The test report shall contain the following information:

- (a) All details necessary for identification of the sample.
- (b) Reference to this Australian Standard, i.e. AS 2300.1.6.
- (c) The pH of the sample to the nearest 0.01 pH unit.
- (d) The temperature at which the pH was measured.
- (e) Details of any dilution of the sample.
- (f) Where applicable, a statement concerning the estimated uncertainty of measurement.
- (g) Date on which the test was performed.
- (h) Any circumstances or unusual observations made during the course of the test which may have had an effect on the result.