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### **NORMES POUR LE LAIT SEC — DETERMINATION DE L'ACIDITE TITRABLE (METHODE PRATIQUE)**

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B.P.: 14966 Yaoundé – CAMEROUN – Tél: 6992 257 777 /Fax.: (237) 222 22 64 96  
E-mail : [enquetepublique@anor.cm](mailto:enquetepublique@anor.cm) – [www.anor.cm/enquetes-publiques](http://www.anor.cm/enquetes-publiques)

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# International Standard



# 6092

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## **Dried milk — Determination of titratable acidity (Routine method)**

*Lait sec — Détermination de l'acidité titrable (Méthode pratique)*

**First edition — 1980-08-15**

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**Descriptors :** dairy products, milk, dried milk, chemical analysis, acidity, volumetric analysis.

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6092 was developed by Technical Committee ISO/TC 34, *Agricultural food products*, and was circulated to the member bodies in June 1977.

It has been approved by the member bodies of the following countries :

|                     |                |                       |
|---------------------|----------------|-----------------------|
| Australia           | India          | Portugal              |
| Austria             | Iran           | Romania               |
| Belgium             | Ireland        | South Africa, Rep. of |
| Bulgaria            | Israel         | Spain                 |
| Canada              | Kenya          | Thailand              |
| Czechoslovakia      | Korea, Rep. of | Turkey                |
| Egypt, Arab Rep. of | Mexico         | United Kingdom        |
| Ethiopia            | Netherlands    | USSR                  |
| France              | New Zealand    | Yugoslavia            |
| Germany, F. R.      | Philippines    |                       |

The member body of the following country expressed disapproval of the document on technical grounds :

Hungary

NOTE — The method specified in this International Standard has been developed jointly with the IDF (International Dairy Federation) and the AOAC (Association of Official Analytical Chemists, USA).

# Dried milk — Determination of titratable acidity (Routine method)

## 1 Scope and field of application

This International Standard specifies a routine method for the determination of the titratable acidity of all types of dried milk.

## 2 References

ISO/R 707, *Milk and milk products — Sampling*.

ISO/R 1736, *Dried milk — Determination of fat content (Reference method)*.

ISO 6091, *Dried milk — Determination of titratable acidity (Reference method)*.

## 3 Definition

**titratable acidity of dried milk** : The number of millilitres of 0,1 mol/l sodium hydroxide solution required to neutralize, in the presence of phenolphthalein, a quantity of the reconstituted milk corresponding to 10 g of solids-not-fat, until the apparition of a pink coloration.

## 4 Principle

Preparation of reconstituted milk by addition of water to a test portion of dried milk corresponding accurately to 5 g of solids-not-fat. Titration with 0,1 mol/l sodium hydroxide solution using phenolphthalein as indicator and cobalt(II) sulphate as reference colour solution. Multiplication of the number of millilitres used in the titration by the factor 2, in order to obtain the number of millilitres in terms of 10 g of solids-not-fat.

The amount of sodium hydroxide solution required is a function of the amount of natural buffering substances present in the product, and of developed or added acid or alkaline substances.

## 5 Reagents

All reagents shall be of recognized analytical quality. Water shall be distilled or deionized water, freed from carbon dioxide by boiling for 10 min before use.

**5.1 Sodium hydroxide**, standard volumetric solution,  $c(\text{NaOH}) = 0,1 \pm 0,000\,2 \text{ mol/l}$ .<sup>1)</sup>

**5.2 Reference colour solution.**

Dissolve 3 g of cobalt(II) sulphate heptahydrate ( $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ ) in water and make up to 100 ml.

**5.3 Phenolphthalein solution.**

Dissolve 2 g of phenolphthalein in 75 ml of 95 % (V/V) ethanol and add 20 ml of water. Add the sodium hydroxide solution (5.1) until 1 drop gives a faint pink coloration, and make up to 100 ml of water.

## 6 Apparatus

**6.1 Analytical balance.**

**6.2 Burette**, graduated in 0,1 ml and with an accuracy of 0,05 ml.

**6.3 Pipettes**, of capacity 2 ml.

**6.4 Measuring cylinders**, of capacity 50 ml.

**6.5 Conical flasks**, of capacity 100 or 150 ml, with ground necks and ground glass stoppers.

1) Hitherto expressed as "0,1  $\pm$  0,000 2 N standard volumetric solution".

## 7 Sampling

See ISO/R 707.

## 8 Procedure

### 8.1 Preparation of the test sample

Transfer the sample to a clean, dry container (provided with an air-tight lid) of capacity about twice the volume of the sample.

Close the container immediately, and thoroughly mix the contents by repeatedly shaking and inverting the container. During these operations, exposure of the sample to the atmosphere should be avoided as far as possible, to minimize adsorption of water.

### 8.2 Test portion

Weigh  $(500/a) \pm 0,01$  g of the test sample (8.1) into each of two conical flasks (6.5),  $a$  being the solids-not-fat content of the sample, expressed as a percentage to two decimal places.

NOTE — The solids-not-fat content of the sample may be calculated by subtracting the fat content (determined in accordance with ISO/R 1736) and the moisture content<sup>1)</sup> from 100.

### 8.3 Determination

**8.3.1** Prepare reconstituted milk by adding 50 ml of water at about 20 °C to the test portion (8.2) and agitating vigorously. Allow to stand for about 20 min.

**8.3.2** Add to one of the conical flasks 2 ml of the reference colour solution (5.2) to obtain a colour standard, and mix by slight swirling.

If a series of determinations on similar products is to be carried out, this colour standard may be used throughout. However it should be discarded after 2 h.

**8.3.3** Add 2 ml of the phenolphthalein solution (5.3) to the second conical flask, and mix by slight swirling.

**8.3.4** Titrate the contents of the second conical flask, while

swirling, by adding the sodium hydroxide solution (5.1) from the burette (6.2) until a faint pink colour similar to that of the colour standard persists for about 5 s. The titration should be completed within 45 s.

Record the volume, in millilitres, of sodium hydroxide solution used, to the nearest 0,05 ml.

## 9 Expression of results

### 9.1 Method of calculation and formula

The titratable acidity is equal to

$$2 \times V$$

where  $V$  is the volume, in millilitres, of the sodium hydroxide solution (5.1) used for titration (8.3.4).

Express the result to one decimal place.

### 9.2 Repeatability

The difference between the results of two determinations carried out simultaneously or in rapid succession by the same analyst shall not exceed 0,4 ml of 0,1 mol/l sodium hydroxide solution per 10 g of solids-not-fat.

## 10 Comparison with the reference method

Verify at intervals whether this routine method gives results in adequate agreement with those obtained using the reference method specified in ISO 6091.

## 11 Test report

The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The test report shall include all details required for the complete identification of the sample.

1) A method for the determination of moisture content is specified in IDF Standard No. 26, *Determination of the water content of dried milk*, which is currently under revision by a joint ISO/IDF/AOAC group of experts.