

MAFF VALIDATED METHODS FOR THE ANALYSIS OF FOODSTUFFS

No.V5

MOISTURE IN COCOA AND CHOCOLATE PRODUCTS

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1. Scope and Field of Application

The method allows the determination of the moisture content of cocoa mass and powder.

2. Definition

Moisture content: the content of total water as determined by the method specified.

3. Principle

The sample is mixed with sand and dried to constant weight at $103 \pm 2^\circ\text{C}$.

4. Reagents

Wherever the use of water is required, distilled or water of equivalent purity is to be used.

4.1 Sea Sand The size of grains must not exceed 0.5 mm diameter. Before use, clean the sand by preparing a slurry of the sand with 10% hydrochloric acid and then washing well with water until a neutral washing is obtained. Dry and ignite the sand at $575 \pm 25^\circ\text{C}$ for 30 min. After ignition allow to cool in a desiccator and, when cool, store in a tightly sealed bottle. Commercially prepared acid- washed sand is also suitable.

5. Apparatus

5.1 Metal weighing dishes, resistant to attack by the sample and the conditions of the test, of minimum diameter 50 mm and minimum height 20 mm, with closely fitting lids.

5.2 Glass rods, of such a length that they lie wholly in the dishes.

5.3 Drying oven, temperature-controlled by thermostat at $103 \pm 2^\circ\text{C}$ throughout the volume of the oven.

5.4 Desiccator, containing freshly activated silica gel (with a water content indicator) or an equivalent desiccant.

5.5 Analytical balance

6. Procedure

6.1 Preparation of the dish

Introduce about 20 g of the treated sand into a weighing dish together with a small glass rod. Place the rod, dish and the upturned lid in the drying oven and dry for 4 hr. at $103 \pm 2^\circ\text{C}$.

Remove the dish and the upturned lid from the oven, immediately place the lid on the dish and the glass rod on top of the lid and then put into the desiccator. Allow the dish to cool to ambient temperature (normally about 1 hr. cooling). Weigh the dish, lid and rod to the nearest 0.2 mg (m_0).

6.2 Test portion

Weigh into the dish, to the nearest 0.2 mg, about 5 g of grated, powdered or otherwise finely divided sample (m_1 = weight of sample, dish, lid and rod). Mix the sample with the sand using the glass rod to achieve as homogeneous a mixture as possible.

Place the dish, contents (including rod) and lid (placed beside the corresponding dish) in the drying oven and dry for 4 hr. at $103 \pm 2^\circ\text{C}$. Remove the dish and lid from the oven, immediately place the lid on the dish and put the covered dish in the desiccator. Allow to cool to ambient temperature and weigh to 0.2 mg. Repeat the drying process, but leave for only 30 min. Repeat until the difference between two successive weighings is no greater than 5 mg (m_2).

7. COSHH

Analysts are reminded that appropriate hazard and risk assessments required by the Control of Substances Hazardous to Health Regulations, 1988 (See "Control of Substances Hazardous to Health - Approved Code of Practice, Control of Substances Hazardous to Health Regulations, 1988") must be made before using this method.

8. Expression of Results

The moisture content, calculated as a percentage by mass of the prepared sample, is given by:

$$\% \text{ moisture content} = \frac{m_1 - m_2}{m_1 - m_0} \times 100$$

where:

m_0 is the weight (mg) at ambient temperature of the dish, sand, lid and rod after drying in the oven.

m_1 is the weight (mg) of the sample, dish, sand, lid and rod before drying.

m_2 is the weight (mg) of the sample, dish, sand, lid and rod after drying in the oven.

9. References

- 9.1 HJ Judd, MD Percival and R Wood, J. Assoc. Publ. Analysts, 1984, **22**, 81-101.
9.2 "Determination of Moisture", International Office of Cocoa and Chocolate/International Sugar Confectionery Manufacturers' Association (IOCC/ISCMA), Analytical Method 3/1952 (previously OICC Analytical Method 3/1952). London.
9.3 Ministry of Agriculture, Fisheries and Food, Food Safety Directorate, MAFF Validated Methods for the Analysis of Food, Introduction, General Considerations and Analytical Quality Control, J. Assoc. Publ. Analysts, 1992, **28**, 11-16

APPENDIX 1

Analytical Quality Control

General principles of analytical quality control are outlined in protocol V0 of the series⁽³⁾.

A1 Repeatability

The absolute difference between two test results obtained under repeatability conditions should not be greater than the repeatability, r , deduced from the collaborative trial data summarised in Tables 1 and 2. For both types of sample, r may be taken to be 0.4% moisture. This precision corresponds to an overall relative standard deviation of repeatability (coefficient of variance of repeatability), RSD_r , of about 4% for the cocoa powder, and about 8% for the drier cocoa mass.

A2 Reproducibility

The absolute difference between two test results carried out under reproducibility conditions should not be greater than the reproducibility, R , deduced from the collaborative trial data summarised in Tables 1 and 2. R may be taken as 0.7% moisture in the cocoa mass samples. In cocoa powder samples its observed value is about twice this, which is unexpectedly high.

A3 Trueness (Bias)

The collaborative trial established satisfactory precision parameters for the method, but its accuracy was not tested by spiking with known amounts of water. However, there is no reason to suspect any systematic bias.

A4 Limit of Detection

This limit has not been established, but the collaborative trial data suggests an accuracy which, if maintained, corresponds to an extrapolated lower limit of roughly 0.4% moisture for a single determination.

A5 Statistical Data Derived from the Results of Interlaboratory Tests

Participants in the collaborative trial each analysed six samples of cocoa powder once (three samples in blind duplicate). All these samples had

been alkali-treated and their cocoa butter content ranged from 11% to 21% by mass.

Participants also each analysed six samples of cocoa mass once (three samples in blind duplicate). Each pair of samples had been tempered and moulded into approximately 50 g size bars; only one pair (O/R) had been alkali-treated. The fat content was 56 - 57% by mass.

Tables 1 and 2 summarise the statistical data; the moisture levels were expressed as a percentage by mass of the sample.

A6 Key to Tables 1 and 2

Symbol	Definition
$\bar{x}$	Overall mean value
S_r	The standard deviation of repeatability
RSD_r	The relative standard deviation of repeatability, expressed as a percentage of the mean (coefficient of variance of repeatability CV_r)
r	Repeatability
S_R	The standard deviation of reproducibility
RSD_R	The relative standard deviation of reproducibility, expressed as a percentage of the mean (coefficient of variance of reproducibility CV_R)
R	Reproducibility

TABLE 1
Statistical Analysis of the % Moisture in Cocoa Powder Samples

Sample	G/M	H/L	J/K
Number of Laboratories retained after eliminating outliers	17	18	17
Number of Laboratories eliminated as outliers	1	0	1
Number of Results accepted after eliminating Outliers	34	36	34
LEVEL OF ANALYTE			
Mean observed value $\bar{x}$	3.12	3.37	3.91
REPEATABILITY			
Standard Deviation S_r	0.14	0.075	0.11
Relative Standard Deviation RSD_r (%)	4.5	2.2	2.8
Repeatability r [$2.8 \times S_r$]	0.39	0.21	0.30
REPRODUCIBILITY			
Standard Deviation S_R	0.49	0.42	0.52
Relative Standard Deviation RSD_R (%)	15.7	12.5	13.3
Reproducibility R [$2.8 \times S_R$]	1.36	1.18	1.45

TABLE 2
Statistical Analysis of the % Moisture in Cocoa Mass Samples

Sample	N/S	O/R	P/Q
Number of Laboratories retained after eliminating outliers	15	15	15
Number of Laboratories eliminated as outliers	3	3	3
Number of results accepted after eliminating Outliers	30	30	30
LEVEL OF ANALYTE			
Mean observed value $\bar{x}$	1.62	1.95	1.72
REPEATABILITY			
Standard Deviation S_r	0.143	0.075	0.09
Relative Standard Deviation $RSD_r(\%)$	8.8	3.8	5
Repeatability $r [2.8 \times S_r]$	0.40	0.21	0.24
REPRODUCIBILITY			
Standard Deviation S_R	0.164	0.207	0.246
Relative Standard Deviation $RSD_R(\%)$	10	11	14
Reproducibility $R [2.8 \times S_R]$	0.46	0.58	0.69

