

MAFF VALIDATED METHODS FOR THE ANALYSIS OF FOODSTUFFS

No. V 21

MOISTURE IN HONEY

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1. Scope and Field of Application

The method allows the determination of the moisture content of clear and normally coloured honeys. It is the same in principle as that described in CAC/12-1969, Codex Alimentarius Commission Recommended European Regional Standard for Honey.

2. Definition

Moisture content: the content of moisture as determined by the method specified.

3. Principle

The refractive index of a test portion is determined at 20°C and converted into moisture content by reference to tables showing concentration as a function of refractive index.

4. Reagents

None.

5. Apparatus

5.1 Refractometer, capable of being read to unity in the fourth decimal place over the refractive index range 1.4700 to 1.5100, provided with means for the circulation of water about the prisms and a thermometer, the bulb of which is immersed in the circulating water stream. The thermometer shall have a certificate of accuracy at 20°C.

5.2 Light source, for the refractometer (5.1) consisting of a sodium lamp of the type recommended by, and adjusted in accordance with the instructions of, the manufacturer of the refractometer.

5.3 Water bath, controlled by a thermostat at 20°C with a differential no larger than 0.5°C, fitted with a pump for circulating water about the prisms of the refractometer (4.1).

5.4 Glass or plastic rod, with an angled, flattened end, as required for applying the test portion to the prism of the refractometer (4.1).

6. Procedure

6.1 Preparation of the sample for analysis

The mass of the sample presented to the laboratory for analysis shall be at least 200 g. The prepared sample shall always be kept in an air-tight and moisture-tight container.

6.1.1 Liquid honey or pressed honey

If the sample is free from granulation, mix carefully by stirring or shaking. If the honey is granular, place in a closed container on a water bath, taking care not to immerse it, and heat for 30 min. at 60°C. If necessary, further heat at 65°C until liquefaction is complete, occasionally shaking the container. Mix carefully and allow to cool rapidly as soon as the sample liquefies. Do not heat honey which has to be used for the determination of hydroxymethylfurfural content or diastase activity. If the honey contains foreign matter (eg wax, twigs, bees or particles of honeycombs), heat the sample to 40°C on a waterbath and strain the honey through cheesecloth in a hot-water jacketed funnel before sample preparation.

6.1.2 Comb honey

Remove the upper part of the combs, if they are sealed. Completely separate the honey from the combs by passing through a sieve; the mesh of the sieve is formed by wires woven to form square apertures of side 0.50 mm. If part of the wax or comb passes through the sieve, heat the sample as described under 6.1.1 and strain the honey through a filter. If the honey is granular, heat it until the wax liquefies, stir, allow to cool and remove the wax.

6.2 Analysis of the prepared sample

Measure the refractive index of the prepared sample at 20°C in the refractometer (5.1).

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

8.1 Calculation

Calculate the moisture content using the refractive index values shown in the Conversion Table below. The moisture is expressed as a percentage by mass of the prepared sample.

8.2 Corrections

The following correction to the refractometer reading must be used if a temperature other than 20°C is employed.

8.2.1 Temperature above 20°C: add 0.00023 per °C.

8.2.2 Temperature below 20°C: subtract 0.00023 per °C.

9. References

- 9.1** DW Lord, MJ Scotter, AD Whittaker and R Wood, J. Assoc. Publ. Analysts, 1989, **26**, 51-76.
- 9.2** 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

Conversion Table for Estimation of Moisture Content

Refractive index (20°C)	Moisture content %	Refractive index (20°C)	Moisture content %
1.5044	13.0	1.4885	19.2
1.5038	13.2	1.4880	19.4
1.5033	13.4	1.4875	19.6
1.5028	13.6	1.4870	19.8
1.5023	13.8	1.4865	20.0
1.5018	14.0	1.4860	20.2
1.5012	14.2	1.4855	20.4
1.5007	14.4	1.4850	20.6
1.5002	14.6	1.4845	20.8
1.4997	14.8	1.4840	21.0
1.4992	15.0	1.4835	21.2
1.4987	15.2	1.4830	21.4
1.4982	15.4	1.4825	21.6
1.4976	15.6	1.4820	21.8
1.4971	15.8	1.4815	22.0
1.4969	16.0	1.4810	22.2
1.4961	16.2	1.4805	22.4
1.4956	16.4	1.4800	22.6
1.4951	16.6	1.4795	22.8
1.4946	16.8	1.4790	23.0
1.4940	17.0	1.4785	23.2
1.4935	17.2	1.4780	23.4
1.4930	17.4	1.4775	23.6
1.4925	17.6	1.4770	23.8
1.4920	17.8	1.4765	24.0
1.4915	18.0	1.4760	24.2
1.4910	18.2	1.4755	24.4
1.4905	18.4	1.4750	24.6
1.4900	18.6	1.4745	24.8
1.4895	18.8	1.4740	25.0
1.4890	19.0		

APPENDIX 2

Analytical Quality Control

General principles of analytical quality control are outlined in protocol V.0 of the series⁽²⁾.

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 below (Table 1). At moisture levels of about 17%, r may be taken to be 0.5% moisture. This corresponds to a relative standard deviation of repeatability (coefficient of variance of repeatability), RSD_r , of 1%.

A2 Reproducibility

The absolute difference between two test results obtained under reproducibility conditions should not be greater than the reproducibility, R , deduced from the collaborative trial data summarised below (Table 1). Overall, R may be taken to be 0.9% moisture, corresponding to a relative standard deviation of reproducibility (coefficient of variance of reproducibility), RSD_R , of 2%.

A3 Trueness (Bias)

Accuracy was not tested by spiking samples with known concentrations of water. However, there is no reason to suspect 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 0.3% moisture for a single determination.

A5 Statistical Data Derived from the Results of Interlaboratory Tests

Participants in the collaborative trial each analysed eight samples of honey once (four samples from different countries in blind duplicate). The samples did not require preparation (6.1) before analysis.

Table 1 summarises the statistical data; the levels of moisture are expressed as a percentage by mass of the sample.

TABLE 1
Statistical Analysis of the % Moisture in Honey

Sample	1/7	2/5	3/8	4/6
Number of Laboratories retained after eliminating outliers	18	18	16	15
Number of Laboratories eliminated as outliers	1	1	3	5
Number of results accepted after eliminating outliers	36	36	32	30
LEVEL OF ANALYTE				
Mean observed value $\bar{x}$	15.9	17.8	17.3	16.0
REPEATABILITY				
Standard Deviation S_r	0.1	0.19	0.08	0.06
Relative Standard Deviation $RSD_r(\%)$	0.6	1.1	0.4	0.4
Repeatability $r [2.8 \times S_r]$	0.28	0.53	0.21	0.17
REPRODUCIBILITY				
Standard Deviation S_R	0.33	0.31	0.14	0.20
Relative Standard Deviation $RSD_R(\%)$	2.1	1.7	0.8	1.2
Reproducibility $R [2.8 \times S_R]$	0.92	0.87	0.40	0.55

A6 Key to Table 1

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