MAFF VALIDATED METHODS FOR THE ANALYSIS OF FOODSTUFFS

MAFF VALIDATED METHOD V20 **APRIL 1992**

METHOD FOR ASH IN HONEY`

Also published in the Journal of the Association of Public Analysts, 28, 177-181

Correspondence on the MAFF Validated Methods Series may be sent to Roger Wood, JFSSG, Food Contaminants Division, c/o Institute of Food Research, Norwich Research Park, Colney, Norwich, NR4 7UA

MAFF VALIDATED METHOD V19: METHOD FOR ASH IN HONEY

Correspondence on this method may be sent to Roger Wood, JFSSG, Food Contaminants Division, c/o Institute of Food Research, Norwich Research Park, Colney, Norwich, NR4 7UA

COSHH AND SAFETY CONSIDERATIONS

Analysts are reminded that appropriate hazard and risk assessments required by the Control of Substances Hazardous to Health Regulation, 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.

1. SCOPE AND FIELD OF APPLICATION

The method allows the determination of the ash content of honey; this is taken as a measure of total mineral content. It is the same in principle as that described in CAC/12-1969, Codex Alimentarius Commission Recommended European Regional Standard for Honey.

2. DEFINITION

Ash content: the content of ash as determined by the method specified.

3. PRINCIPLE

The residual mass of a test portion is determined gravimetrically after incineration in an oxidising atmosphere at 600°C and calculated as a percentage by mass of the sample.

4. REAGENTS

All reagents should be of recognised analytical grade unless specified otherwise.

4.1 Olive oil, food grade.

4.2 Dilute hydrochloric acid, approximately 7 g per 100 ml. Carefully add, with stirring, 100 ml of hydrochloric acid (4.2.1) to 500 ml of water and mix.

4.2.1 Hydrochloric acid, concentrated, (HC1, density 1.64 g/ml)

5. APPARARIUS

5.1 Incineration dishes, made of platinum or silica.

5.2 Electric muffle furnace, air-ventilated, temperature controlled by thermostat at 600°C with a differential no larger than 25°C, fitted with a pyrometer.

5.3 Infra-red lamp

5.4 Desiccator, containing an efficient desiccant, eg dried silica gel.

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 200g. 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 liquefication 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 Preparation of the incineration dish

Clean the incineration dish (5.1), whether new or not, with boiling dilute hydrochloric acid (4.2). Rinse it free from acid with large quantities of water. Heat it for 30 min. in the muffle furnace (5.2). Remove it from the furnace, allow it to cool to ambient temperature in the desiccator (5.4) and weigh it to the nearest 0.1 mg (m_1).

6.3 Analysis of the prepared sample

6.3.1 Weigh into the prepared incineration dish (6.2), to the nearest 1 mg, about 5-10 g of honey (m_0).

6.3.2 Place the dish and contents (6.3.1) in the muffle furnace (5.2) and heat gently until the sample becomes black and dry. Care must be taken to avoid risk of loss through foaming and excessive swelling of the mass. An infra-red lamp (5.3) may be used to aid carbonisation of the sample prior to putting it in the muffle furnace; such initial charring may be essential to prevent excessive foaming. The addition of a few drops of olive oil (4.1) may also help to prevent excessive swelling.

6.3.3 Ignite the dish at 600°C until no further apparent change in colour of the residue ash occurs.

6.3.4 Remove the dish from the furnace, place it in the desiccator (5.4) and allow it to cool to ambient temperature.

6.3.5 Weigh the dish and residue to the nearest 0.1 mg.

6.3.6 Repeat operations 6.3.3, 6.3.4 and 6.3.5 until the difference between two successive weighings is less than 0.1 mg. Designate the final weight m_2.

7. EXPRESSION OF RESULTS

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

$$\% \text{ Ash content} = 100 \times (m_2 - m_1)/m_0$$

where;

m_0 is the mass of the test portion, in g (6.3.1);

m_1 is the mass of the prepared incineration dish, in g (6.2);

m_2 is the mass of the incineration dish and the residue, in g (6.3.6).

8. REFERENCES

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

8.2 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
ANALYTICAL QUALITY CONTROL

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

Al 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). Within the range 0.05-0.2% ash, r may be taken as 0.05% ash. This corresponds to a relative standard deviation of repeatability (coefficient of variance of repeatability), RSD_r, of 7-29%. The value of r may be somewhat higher (0.1% ash) at higher ash levels (above 0.2% ash).

A2. REPRODUCIBILIIY

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 0.05-0.14% ash, corresponding to a relative standard deviation of reproducibility (coefficient of variance of reproducibility), RSD_R of 22-40%.

A3. TRUENESS (BIAS)

Accuracy was not tested by spiking samples with known concentrations of minerals. 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 limit of ash content of roughly 0.04% ash 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 ash are expressed as a percentage by mass of the sample.

TABLE 1
STATISTICAL ANALYSIS OF THE % ASH IN HONEY SAMPLES

Sample	1/7	2/5	3/8	4/6
Number of laboratories retained after eliminating outliers	19	17	19	19
Number of laboratories eliminated as outliers	0	2	0	0
Number of accepted results after eliminating outliers	38	34	38	38
LEVEL OF, ANALYTE -				
Mean observed value x	0.23	0.05	0.20	0.08
REPEATABILITY				
Standard deviation S_r	0.03	0.01	0.01	0.02
Relative standard deviation RSD_r (%)	14	29	7	22
Repeatability r [$2.8 \times S_r$]	0.09	0.04	0.04	0.05
REPRODUCIBILITY				
Standard deviation S_R	0.05	0.02	0.05	0.03
Relative standard deviation RSD_R (%)	22	36	23	40
Reproducibility R [$2.8 \times S_R$]	0.14	0.05	0.13	0.09

A6. KEY TO TABLE 1

SYMBOL	DEFINITION
-	
x	Overall observed mean value
Sr	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