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MAFF VALIDATED METHODS FOR THE ANALYSIS OF FOODSTUFFS

No. V15

SOLUBLE SOLIDS IN VINEGAR

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1. Scope and Field of Application

The method determines the loss of mass on drying of vinegar

2. Definition

Total soluble solids: the weight of matter remaining after drying by the method specified.

3. Principle

The residual mass of a test portion is determined after evaporation on a water bath followed by drying at atmospheric pressure in an oven at 103 ± 2°C. To ensure the total volatilisation of the acetic acid, the evaporation step is repeated three times after restoration to at least the original volume with distilled water.

4. Reagents

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

5. Apparatus

5.1 Pipettes, 10 ml.

5.2 Filter and filter papers, slow-filtering paper.

5.3 Dishes, of platinum, ceramic or glass. The dishes must have lids which fit very well but which can be readily removed. The dishes should be 75 mm in diameter.

5.4 Water bath

5.5 Atmospheric pressure drying oven, well ventilated and thermostatically controlled with temperature regulation at 103 ± 2°C. The temperature should be uniform throughout the oven.

5.6 Desiccator, containing freshly activated silica gel with a water content indicator, or equivalent desiccant.

5.7 Analytical balance, capable of weighing to at least 0.1 mg.

5.8 Glass stirring rod

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6. Procedure

6.1 Uncover the dish and place it and its lid in the oven at 103°C for 1 hr.

6.2 Place the lid on the dish and transfer the covered dish to the desiccator.

6.3 Allow the dish to cool to room temperature and accurately weigh to the nearest 0.1 mg (m_1).

6.4 Shake the sample, and filter it through the filter paper.

6.5 Pipette 10 ml of sample into the dish.

6.6 Place the dish on a boiling water bath and evaporate almost to dryness.

6.7 Add 15 ml of distilled water to the dish and stir.

6.8 Wash the stirring rod into the dish with a small quantity of distilled water.

6.9 Evaporate almost to dryness on a boiling water bath.

6.10 Repeat processes 6.7 to 6.9 a further two times.

6.11 Place uncovered dish and its lid in the oven at 103°C for 3 hr.

6.12 Cover the dish and transfer the covered dish to the desiccator.

6.13 Allow the dish to cool to room temperature and accurately weigh to the nearest 0.1 mg as quickly as possible (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

Calculate the total soluble solids of the sample, expressed as a percentage mass to volume, by the formula:

$$\% \text{ Total soluble solids} = (m_2 - m_1) \times 10$$

where

m_1 is the weight in grams of the empty dish and lid after process 6.3;

m_2 is the weight in grams of the dish, its lid and the final dried sample after process 6.13.

9. References

9.1 MJ Scotter and R Wood, J. Assoc. Publ. Analysts, 1985, **23**, 107-117.

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

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 in Table 1. For untreated vinegar (and for the fortified vinegars), r may be taken to be 0.10 g per 100 ml; this corresponds to a relative standard deviation of repeatability (coefficient of variance of repeatability), RSD_r, of about 4%.

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 Table 1.
R may be taken as 0.17 g per 100 ml, which corresponds to a relative standard deviation of reproducibility (coefficient of variance of reproducibility), RSD_R, of about 8%.

A3 Trueness (Bias)

The results of the collaborative trial demonstrate that the presence of additional substances in the stock vinegar does not affect the performance of the procedure. In particular, added acetic acid is removed quantitatively during the drying process, while added sodium chloride and added citric acid is recovered quantitatively. The extent of any systematic bias due to the occlusion of acetic acid in the soluble solids residue obtained after drying remains unknown, but is unlikely to be significant; in any case it is demonstrably less than that associated with the corresponding AOAC procedure, which was also tested during the collaborative trial.

A4 Limit of Detection

This limit has not been established, but the collaborative trial data suggest an accuracy which, if maintained, corresponds to an extrapolated lower limit of roughly 0.1 g per 100 ml for a single determination.

A5 Statistical Data Derived from the Results of Interlaboratory Tests

Participants in the collaborative trial each analysed sixteen subsamples of vinegar once (eight samples in blind duplicate). Sample 2/10 was untreated vinegar; the other samples were the same vinegar containing in

addition 2 g glacial acetic acid per 100 ml (A), 0.5g sodium chloride per 100 ml (S) and/or 0.5 g citric acid per 100 ml (C), as follows: sample 4/6, A; sample 8/11, S; sample 1/16, C; sample 3/15, A+S; sample 5/13, A+C; sample 7/9, S+C; sample 12/14, A+S+C.

Table 1 summarises the statistical data, expressed as g soluble solids per 100 ml vinegar.

TABLE 1

Statistical Analysis of the % Soluble Solids in Vinegar Samples

Sample	2/10	4/6	8/1	11/16	3/15	5/13	7/9	12/14
Number of Laboratories retained after eliminating outliers	18	18	17	18	15	18	18	18
Number of Laboratories eliminated as outliers	0	0	1	0	3	0	0	0
Number of results accepted after eliminating outliers	36	36	34	36	30	36	36	36
LEVEL OF ANALYTE								
Mean observed value $\bar{x}$	0.77	0.75	1.27	1.22	1.25	1.20	1.72	1.70
REPEATABILITY								
Standard Deviation S_r	0.03	0.02	0.02	0.03	0.02	0.06	0.03	0.04
Relative Standard Deviation RSD_r(%)	4	3	2	2	2	5	2	2
Repeatability r [2.8 x S_r]	0.09	0.06	0.05	0.08	0.06	0.16	0.07	0.11
REPRODUCIBILITY								
Standard Deviation S_R	0.06	0.06	0.05	0.06	0.05	0.08	0.05	0.07
Relative Standard Deviation RSD_R(%)	8	8	4	5	4	7	3	4
Reproducibility R [2.8 x S_R]	0.17	0.18	0.14	0.16	0.15	0.22	0.15	0.19

A7 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