

MAFF VALIDATED METHODS FOR THE ANALYSIS OF FOODSTUFFS

No. V 24

INSOLUBLE MATTER IN INSTANT COFFEE

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1. Scope and Field of Application

The method allows the determination of the water-insoluble matter content of instant coffee.

The precision of the method is acceptable at levels above 50 mg of insoluble matter per 100 g of sample (i.e. 0.05% *m/m*); however, the "natural" levels of insoluble matter in instant coffees should be considerably less than this concentration. The method will therefore have limited application as a statutory procedure, though it will discriminate against gross adulteration of samples by insoluble material.

2. Definition

Insoluble matter content: the content of the water-insoluble matter as determined by the method specified.

3. Principle

Test portions of the sample are dissolved in water and filtered through a specified filtering disc with square apertures of 100 $\mu\text{m} \times 100 \mu\text{m}$; the residue trapped by the disc is dried and determined gravimetrically.

4. Reagents

None.

5. Apparatus

5.1 Filtration apparatus, illustrated in Fig. 1 and consisting of the following parts. Apparatus 5.1.2 to 5.1.5 is not specified exactly, but it is essential that a sieve conforming to the specifications given in 5.1.1 is always employed.

5.1.1 Metallic filtering disc, micro-precision sieve, made from nickel by electroforming, with support screen: diameter 29 mm, massive periphery ca. 3 mm wide, 70 μm thick, square holes of side 100 μm , 159 mesh.

5.1.2 Borosilicate glass cup

5.1.3 Borosilicate glass tulip

5.1.4 Flange for heading joint

- 5.1.5 Sealing rings
- 5.2 Tweezers, for microscope slides.
- 5.3 Precision balance, reading to 0.01 mg.
- 5.4 Filtering installation, for reduced pressure filtration.
- 5.5 Oven, without forced ventilation, thermostatically controlled at $103 \pm 2^\circ\text{C}$.
- 5.6 Desiccator, containing freshly activated silica gel (or an equivalent desiccant) with a moisture content indicator.
- 5.7 Analytical balance

6. Procedure

6.1 Preparation of the filtration apparatus

Spread glass beads (diameter about 4 mm) all over the bottom of a small petri dish (diameter about 60 mm). Place the clean filter disc into the petri dish and dry for 30 min. in an oven at $103 \pm 2^\circ\text{C}$. Cool to room temperature in a desiccator and weigh the disc to the nearest 0.1 mg (M_0).

Set up the filtration apparatus according to Fig. 1, and fix it onto the filtering flask by means of a rubber joint.

6.2 Preparation of the solution

Accurately weigh, to the nearest 1 mg, about 5 g (M_1) of sample into an 800 ml beaker. Add 500 ml of boiling water, and stir for 10-15 sec. with a glass rod with rounded ends.

6.3 Filtration

Pour the solution as hot as possible into the filtration apparatus. Filter at room pressure; use of vacuum does not aid filtration. Pat the top of the cup with the palm of the hand to facilitate the filtration.

Rinse the beaker and the cup inside with about 200 ml of hot water, so that all insoluble particles are collected on the filtering disc. Finally, apply vacuum to remove the water held back on the disc.

Carefully dismantle the filtration apparatus in order to take out the filtering disc without losing insoluble matter. Place the filtering disc into the petri dish and dry it for 30 min. in an oven at $103 \pm 2^\circ\text{C}$. Allow it to cool to room temperature in a desiccator and weigh the disc to the nearest 0.1 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 content of insoluble matter, calculated as a percentage by mass of the prepared sample, is given by:

$$\% (m/m) \text{ Insoluble matter} = 100 \times (M_2 - M_0) / M_1$$

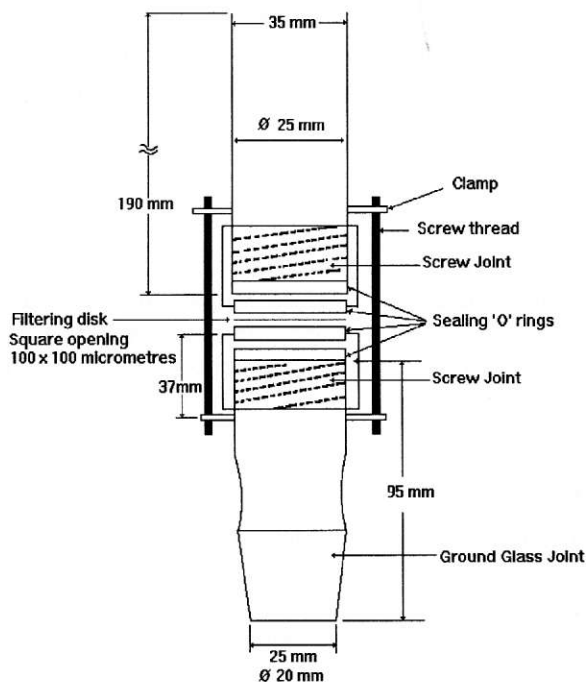
where:

- M_0 is the mass of the clean, dry sieve disc, in g (6.1);
- M_1 is the mass of the test portion, in g (6.2);
- M_2 is the mass of the sieve disc and insoluble matter, in g (6.3).

9. References

- 9.1 SL Reynolds, SA Thorpe and R Wood, J. Assoc. Publ. Analysts, 1983, 21, 47-52.
- 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.

Fig. 1: Diagram of the filter apparatus used in the trial.



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 below (Table 1). For instance, at levels of insoluble matter corresponding to sample C (i.e. 5 mg of insoluble matter in a 5 g sample, or 0.1% m/m), r may be taken as 2 mg; this corresponds to a relative standard deviation of repeatability (coefficient of variance of repeatability), RSD_r , of 15%. This precision may be expected to deteriorate at lower levels, and the method is suitable as a statutory procedure only when samples with concentrations of insoluble matter in excess of 0.05% m/m are analysed.

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). For instance, at levels of insoluble matter corresponding to sample C (i.e. 5 mg of insoluble matter in a 5 g sample, or 0.1% m/m), R may be taken to be 2 mg; this corresponds to a relative standard deviation (coefficient of variance), RSD_R of 16%. This precision may be expected to deteriorate at lower levels.

A3. Trueness (Bias)

The observed levels of insoluble matter obtained during the collaborative trial were lower than the expected values; at 5 mg of insoluble matter (0.1% m/m), the recovery was 85%, and over 90% at higher levels. There may therefore be a systematic bias, perhaps due to the loss of insoluble material adhering to the filter sieve clamping apparatus.

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 insoluble matter content of roughly 0.5 mg of insoluble matter (0.01% m/m) for a single determination.

A5. Statistical Data Derived from the Results of Interlaboratory Tests

Participants in the collaborative trial each analysed ten samples of instant coffee once (five samples in duplicate). Each sample weighed about 5 g, and the whole of it was taken for analysis. Sample A was instant coffee; samples B- E were spiked with known weights of insoluble matter (see Table 1, 'Expected "true" value'). This added insoluble matter consisted of coffee grounds that had been exhaustively extracted with boiling water, filtered and dried.

Table 1 summarises the statistical data; the levels of analyte are expressed as mg of insoluble matter per 5 g sample.

TABLE 1
The Statistical Analysis of Insoluble Matter (mg/5g)
in Instant Coffee Samples

Sample	A	B	C	D	E
Number of Laboratories retained after eliminating outliers	14	15	16	15	16
Number of Laboratories eliminated as outliers	2	1	0	1	0
Number of results accepted after eliminating outliers	28	30	31	30	32
LEVEL OF ANALYTE					
Mean observed value $\bar{x}$	0.28	1.04	4.53	9.45	28.67
Expected "True" Value	A	1.0+A	5.0+A	10.0+A	30.0+A
Recovery (100% = quantitative) (%)		76	85	92	95
REPEATABILITY					
Standard Deviation S_r	0.18	0.25	0.69	0.70	0.89
Relative Standard Deviation $RSD_r(\%)$	62	24	15	7.4	3.1
Repeatability r [$2.8 \times S_r$]	0.49	0.70	1.93	1.95	2.50
REPRODUCIBILITY					
Standard Deviation S_R	0.25	0.32	0.72	1.01	1.34
Relative Standard Deviation $RSD_R(\%)$	89	31	16	11	4.7
Reproducibility R [$2.8 \times S_R$]	0.70	0.90	2.03	2.84	3.76

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