

1ST WORKING DRAFT : 1990-1-10

**Cigarettes - Determination of water in smoke condensates  
Karl Fischer method**

**0 Introduction**

The revision of ISO 4387 based upon the work and conclusions of Task Forces expressed in the Recommended Method No. 23 of CORESTA (Cooperation Centre for Scientific Research Relative to Tobacco) specifies the use of gas chromatography for the determination of water in smoke condensate solutions. Whereas this recommendation is undoubtedly correct, it is realised that not all laboratories have access to an appropriate chromatograph. Accordingly, this International Standard has been produced to specify an appropriate water analysis using Karl Fischer reagents.

**1 Scope**

This International Standard specifies the use of the Karl Fischer method for the determination of water in cigarette smoke condensate.

**2 Principle**

The smoke condensate from the mainstream smoke is dissolved in a solvent. The water content of an aliquot of the solution is determined by titration with standardised Karl Fischer reagent.

**3 Reagents**

All reagents used shall be of analytical reagent quality.

**3.1 Karl Fischer reagent (KFR)** with a water equivalent between 3,5 and 4,5 mg/ml.

**3.2 Methanol** with a water content of less than 0,05g/100g.

**3.3 Silica gel**, freshly activated.

**4 Apparatus**

Usual laboratory apparatus and the following items:

NOTE - The use of automatic apparatus is recommended for the pipetting and titration of the solvents and solutions.

**4.1 Karl Fischer apparatus** for automatic titration, with automatic burettes of 25 ml capacity each, graduated in 0,05 ml, provided with connecting pieces and tubes, and one additional automatic burette of 50 ml capacity.

**4.2 Sleeve caps** with conical 29/32 ground-glass joints and taps.

**4.3 Conical stopper** 29/32, made of PTFE, provided with a ventilation piece.

**4.4 Micro-syringe**, capacity 50  $\mu$ l.

4.5 Conical flasks with a conical 29/32 ground glass joint, capacity 150 cm<sup>3</sup> or 250 cm<sup>3</sup> (see 5.3).

## 5 Procedure

Care shall be taken during all operations to avoid contamination from atmospheric moisture.

All glassware used in the water determination shall be heated at 105°C for at least 1 h after visible water has evaporated, and cooled and stored in a desiccator over silica gel until used.

### 5.1 Standardization of Karl Fischer reagent

Transfer, by means of an automatic burette, about 25 ml of methanol into the titration vessel and titrate automatically with Karl Fischer reagent. Add, by means of a micro-syringe, 50 µl of water ( $V_w$ ) and titrate again with Karl Fischer reagent ( $V_t$ ). Carry out this procedure three times and calculate the mean value ( $\bar{V}_t$ ). Repeat the standardization of Karl Fischer reagent on every working day.

### 5.2 Calculation of water equivalent

The water equivalent  $E$  of the Karl Fischer reagent, expressed in milligrams H<sub>2</sub>O per millilitre, is given by the equation

$$E = \frac{m_w}{\bar{V}_t}$$

where

$m_w$  is the mass, in milligrams, of the volume of water ( $V_w$ ) used for the standardization of the Karl Fischer reagent;

$\bar{V}_t$  is the mean volume, in millilitres, of the Karl Fischer reagent used for the titration of the water.

### 5.3 Test Portion

Collect the cigarette smoke condensate according to ISO/DIS 4387:1990 on a routine analytical cigarette-smoking machine complying with the requirements of ISO/DIS 3308:1990.

Wearing gloves, remove the sealing devices from the smoke trap, open it and remove the filter disc with forceps. Fold it twice, total particulate matter inwards, being careful to handle only the edge with forceps and gloved fingers. Place the folded disc in a dry conical flask (maximum 150 cm<sup>3</sup> for 44 mm discs, maximum 250 cm<sup>3</sup> for 92 mm discs).

Wipe the inner surface of the filter holder front with two separate quarters of an unused conditioned filter disc and add these to the flask.

Run a maximum of two portions of 20 ml of methanol into the flask. The folded disc shall be covered with solvent. In the case of a 92 mm glass fibre filter trap, 80 ml of methanol is necessary.

Stopper the flask immediately and shake gently on an electric shaker for at least 20 minutes ensuring that the disc does not disintegrate.

#### 5.4 Determination

Measure the water content of this solution by injecting a suitably sized aliquot (e.g. 10 ml) into the titration vessel.

Follow the same procedure with each of the blank smoke traps used for the determination of water.

### 6 Calculation and expression of results

#### 6.1 Method of Calculation

The water content  $W$  of the total particulate matter for each trap, expressed in milligrams per cigarette, is given by the equation

$$W = \frac{(V - V_b) \times E \times V_k}{q \times V_a}$$

where

$V$  is the volume, in millilitres, of the Karl Fischer reagent used for the titration of the total particulate matter solution;

$V_b$  is the volume, in millilitres, of the Karl Fischer reagent used for the blank titration;

$E$  is the water equivalent of the Karl Fischer reagent, in milligrams  $H_2O$  per millilitre;

$q$  is the number of cigarettes smoked into each trap;

$V_k$  is the volume, in millilitres, of methanol used for dissolving the total particulate matter;

$V_a$  is the volume, in millilitres, of the total particulate matter solution used for the titration.

#### 6.1.2 Mean water content of the smoke condensate per set of smoking runs

Calculate the mean of the results obtained for each trap per set of smoking runs.

**6.1.3 Mean water content of the smoke condensate for the whole test sample**

Calculate the mean of the results obtained for each smoking run.

**6.2 Expression of results**

Express the test results as follows:

- (a) water content, expressed in milligrams per cigarette smoked, to the nearest 0,01 mg for each individual smoking run;
- (b) water content, expressed in milligrams per cigarette smoked, to the nearest 0,01 mg for each set of smoking runs;
- (c) mean water content, expressed in milligrams per cigarette smoked, to the nearest 0,1 mg for the whole test sample or to the nearest 0,01 mg if the confidence interval is expressed.

**7 Test Report**

7.1 The test report shall show the method used and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The test report shall include all details required for complete identification of the sample. The test report shall, in particular, include the items of information listed in 7.2 to 7.5.

**7.2 Description of the product tested**

**7.3 Sampling procedure**

- (a) method of sampling;
- (b) number of cigarettes of the test sample;
- (c) date and place of purchase or sampling.

7.4 Test results, together with their precision, expressed in accordance with 6.2

**7.5 Date of Test**