

CORESTA RECOMMENDED METHOD N° 59

DETERMINATION OF TRIACETIN IN FILTER RODS BY GAS CHROMATOGRAPHIC ANALYSIS

(June 2004)

1. FIELD OF APPLICATION

The method is applicable to cellulose acetate cigarette filter rods plasticized with triacetin to a maximum concentration of 150 mg triacetin per gram of filter rod and containing no other additives.

2. REFERENCES

ISO 3402:1999

Tobacco and tobacco products – Atmosphere for conditioning and testing.

2.1. Definitions

<i>Bath Ratio:</i>	mg material per ml extraction solvent
<i>Triacetin:</i>	glyceryl triacetate (CAS-number: 102-76-1)
<i>Additive:</i>	flavour, charcoal, other adsorbents or other plasticizers
<i>Rod weight:</i>	mean weight of filter rods as received and used for extraction (including the weight of plug wrap paper, glue and cellulose acetate as well as triacetin)

3. PRINCIPLE

Triacetin is extracted from filter material by shaking in ethanol. The triacetin concentration of this extract is determined by using gas chromatography. Results are expressed as the weight of triacetin per filter rod (mg/rod).

Note: Triacetin percentage can be calculated if the weights of all other rod components are known and quantified.

4. APPARATUS

- 4.1. A gas chromatograph preferably equipped with a flame ionisation detector, (however other appropriate detectors can be used) and with an integration unit (or data handling unit). Analysis can be carried out on packed or capillary columns. A list of suitable GC columns is given in 7.1 and example conditions are shown in Appendix 2.
- 4.2. A standard laboratory flask-shaking machine.
- 4.3. The necessary general laboratory equipment, for the preparation of samples, standards and reagents. All laboratory equipment should be glass.
- 4.4. A suitable conditioning environment to ensure compliance with ISO 3402.

7. PROCEDURE

7.1. Gas Chromatography

Set up and operate the gas chromatograph according to the manufacturer's instructions.

Ensure the solvent, internal standard, triacetin and other component peaks are baseline separated.

Examples of suitable GC columns and conditions are listed below and in Appendix 2:

DB-1 (100% DiMe-Polysiloxane), DB-5 (5% Ph-Polysiloxane), SIL-CP-19 (19% CN-Propyl-Polysiloxane), DB-Wax (PEG).

7.2. Calibration of the Gas Chromatograph

Inject duplicate aliquots of the standard solutions into the gas chromatograph. Record the peak areas of the triacetin and internal standard. Calculate the ratio of the peak areas for triacetin to the internal standard. Establish the calibration graphs between the area ratios and the triacetin concentrations. Calculate the linear regression parameters.

7.3. Calibration check

A full calibration procedure should be carried out prior to sample analysis using the prepared standard solutions described in section 6.

To check calibration use an independently prepared standard after every 10 sample vials. If the value for this solution differs by more than $\pm 3\%$ from the original calibration value the full calibration must be repeated.

7.4. Filter rod sample preparation

Conditioning: Condition the filter rods for a minimum of 24 hours under the environment specified in ISO 3402.

Filter rod preparation: Take five rods and slit them longitudinally. Cut each rod into approximately equal segments of minimum 10 mm and maximum of 20 mm length. Place rod segments including the plug wrap paper in a 250 ml Erlenmeyer flask. Add 100 ml of extraction solvent, as described in section 5.5., with a pipette (or similar precision delivery device). Seal the top of the flask securely.

Extraction: A minimum of two extractions should be carried out. The prepared flask should be placed on a flask shaker for 3 hours. After shaking is complete, transfer aliquots of the solutions to vials for testing on the gas chromatograph.

Note: The bath ratio (as defined in 2.1) based on five uncut rods should be at least in the range 20:1 to 40:1 mg material per ml extraction solvent.

7.5. Measurement and calculation

Inject duplicate aliquots of each vial into the gas chromatograph using the conditions shown in Appendix 2. For each injection record the peak areas of triacetin and of the

internal standard. Calculate the ratio of the peak area of triacetin to that of the internal standard.

Using the calibration procedure produced in section 7.2 determine the concentration of triacetin in the extraction solution as mg/ml. Ensure that values lie within the range of standards prepared in section 6. Calculate the triacetin content in mg/filter rod as described in section 8 and report the mean to the nearest 0.1 mg.

8. CALCULATION OF TRIACETIN

The linear equation of the calibration of the GC (described in section 7.2) should be used for calculating the concentration of triacetin in the extraction solution.

$$C = \frac{Y - B}{A}$$

C: concentration of triacetin in the extraction solution (mg/ml)

Y: ratio of the peak areas of triacetin and internal standard in the chromatogram

B: y-axis intercept of the linear regression line (calibration curve)

A: slope of the linear regression line (calibration curve)

$$\text{mg triacetin per filter rod} = \frac{C \times V}{Z}$$

V: volume of solvent used for extraction (ml)

Z: number of filter rods used for extraction

For informational purposes, the mean weight of the rods may also be reported.

9. REPEATABILITY AND REPRODUCIBILITY

A collaborative experiment in 2003, in which 13 laboratories took part, obtained the following values for the repeatability (r) and reproducibility (R) of the triacetin content of filter rods.

The difference between two single results found on matched filter rod samples by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value (r) on average not more than once in 20 cases in the normal and correct operation of this method.

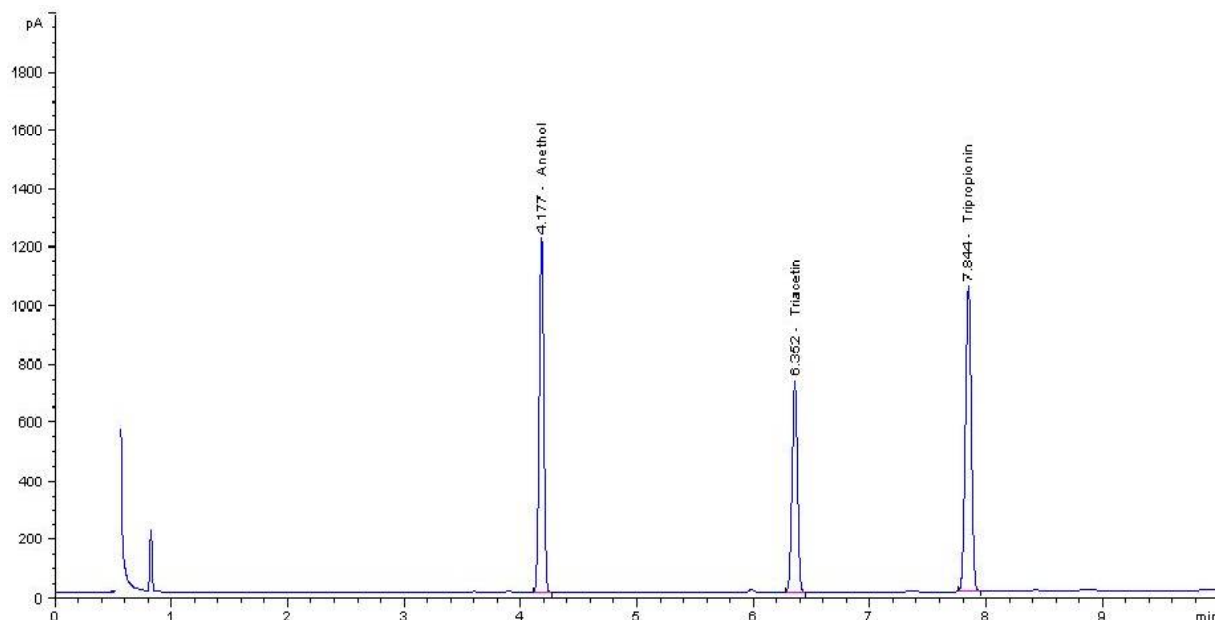
Single results on matched filter rod samples reported by two laboratories will differ by more than the reproducibility (R) on average not more than once in 20 cases in the normal and correct operation of this method.

	Low [~26 mg/rod]	Medium [~50 mg/rod]	High [~74 mg/rod]
Repeatability [r]	1,63	3,49	4,31
Reproducibility [R]	6,00	7,84	15,95

APPENDICES

APPENDIX 1: EXAMPLE CHROMATOGRAM

DB-Wax-Column



APPENDIX 2: EXAMPLE GC-CONDITIONS USING DB-WAX-COLUMN

GC

Standard GC equipped with an autosampler

Injector

Injection Volume 1µl

Inlet

Type Split/Splitless
 Mode Split
 Temperature 250°C
 Pressure 0.97bar
 Gas Type Helium
 Split Ratio 5:1
 Split Flow 87.8ml/min
 Total Flow 108.4ml/min

Inlet liner:

Split liner packed with deactivated fused silica or glass wool according to the GC manufacturers recommendations

Oven

Initial Temperature 120°C
 Initial Time 0 min

Rate 10°C/min
 Final Temperature 230°C
 Final Time 5 min

Detector

Type FID
 Temperature 250°C
 Make-up-Gas Nitrogen
 Make-up-Flow 5 ml/min
 Hydrogen Flow 40 ml/min
 Air Flow 300 ml/min
 Range 0

Column

Type Fused Silica, 30m x 0.53mm i.d.
 Supplier J & W
 Stationary Phase DB-Wax
 Film Thickness 1µm
 Flow 17.6ml/min @ 120°C

Cycle Time

16min.