

CORESTA RECOMMENDED METHOD N° 45

DETERMINATION OF PHOSPHATE IN CIGARETTE PAPER

(January 1998)

1. FIELD OF APPLICATION

The method is applicable to all types of cigarette paper.

2. DEFINITIONS

Phosphates are salts of orthophosphoric acid. Phosphates in cigarette paper influence the ash appearance of a burning cigarette and the burning rate of the cigarette paper and therefore the puff number of the cigarette. Phosphates are usually added to the cigarette paper as disodium hydrogen phosphate or monoammonium dihydrogen phosphate.

3. REFERENCES

ISO 186:1994

Paper and board - Sampling to determine average quality.

ISO 187:1990

Paper, board and pulps - Standard atmosphere for conditioning and testing and procedure for monitoring the atmosphere and conditioning of samples.

ISO 287:1985

Paper and board - Determination of moisture content - Oven-drying method.

ISO 5725-2:1994

Accuracy (trueness and precision) of measurement methods and results -

Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

4. PRINCIPLE

Phosphate ions (PO_4^{3-}) react in an acid solution with ammonium vanadate and ammonium heptamolybdate to form a yellow-coloured molybdenum vanadate phosphoric acid complex which can be measured photometrically.

5. APPARATUS AND MATERIALS

5.1. Apparatus

- analytical balance with an accuracy of 0,001 g;
- UV Spectrophotometer, double beam;
- glass or plastic cuvettes, 10 mm light path;
- micro pipettes;
- pipette 50 cm³;
- calibrated flask 50 cm³ and 100 cm³.

5.2. Materials

- distilled water;
- 1 mol·L⁻¹ hydrochloric acid;
- ashfree filter papers;
- P-reagent.

Note: reagent will deteriorate with time as a result of temperature and radiation. The response should be checked periodically.

It is recommended that test solutions containing ammonium vanadate and ammonium heptamolybdate hereafter referred to as P-reagent are used. Such test kits are available from various suppliers (e.g. E. MERCK, Darmstadt).

6. STANDARD SOLUTIONS

6.1. Preparing the standard solutions for the calibration curve

To calibrate the equipment, standard solutions containing 0, 5, 10, 15 and 20 mg PO₄³⁻ per 50 cm³ of 0,5 mol·L⁻¹ hydrochloric acid are used. Various phosphates can be used, e.g. disodium phosphate, potassium phosphate, monoammonium phosphate or phosphoric acid, all with a purity of p.a.

Weight of phosphate salts for a concentration of 20 mg PO₄³⁻ / 50 cm³

	mol. weight (g/mol)	weight (mg/50 cm ³)
KH ₂ PO ₄	136,09	28,66
K ₃ PO ₄ ·3H ₂ O	266,32	56,09
Na ₃ PO ₄ ·12H ₂ O	380,12	80,05
Na ₂ HPO ₄	141,96	29,90
(NH ₄) ₂ HPO ₄	132,06	27,81
PO ₄ ³⁻	94,97	

Prepare 5 dilutions between 0 and 20 mg PO_4^{3-} in 50 cm^3 in 0,5 mol·L⁻¹ hydrochloric acid equivalent to 0-2% (m/m) of phosphate in cigarette paper. Perform six replicate measurements per calibration point.

(PO_4^{3-}) content in solution (%) (m/m)	(PO_4^{3-}) in cigarette paper (%) (m/m)
0	0
0,01	0,5
0,02	1,0
0,03	1,5
0,04	2,0

Preparation of the blank sample:

Pipette 1 cm^3 P-reagent + 3 cm^3 0,5 mol·L⁻¹ hydrochloric acid into a cuvette.

6.2. Measuring the standard solutions

Pipette 1 cm^3 P-reagent + 2,5 cm^3 0,5 mol·L⁻¹ hydrochloric acid + 0,5 cm^3 standard solution into a cuvette. After approx. 5 minutes the absorbance can be measured on the UV spectrophotometer at a wavelength of 430 nm. Perform six replicate measurements for each calibration point.

7. TEST PROCEDURE

Preparation and measurement of the paper sample.

7.1. Preparation

- weigh approx. 1 g of shredded cigarette paper conditioned according to ISO 187:1990 in a 100 cm^3 Erlenmeyer-flask and correct absorbancy reading of the UV spectrophotometer to 1,000 g of cigarette paper;
- add 50 cm^3 0,5 mol·L⁻¹ hydrochloric acid;
- leave suspension of paper in 0,5 mol·L⁻¹ hydrochloric acid for 20 min, at room temperature (leave to stand, stir or swirl);
- filter the suspension after 20 min. through an ashfree filter paper;
- use the filtrate for the photometric determination of PO_4^{3-} .

7.2. Measurement of the paper sample

Pipette 1 cm^3 P-reagent and **a** cm^3 0,5 mol·L⁻¹ hydrochloric acid and **b** cm^3 of test solution into a cuvette (**a** cm^3 + **b** cm^3 = 3 cm^3) and by using the UV Spectrophotometer measure the absorbance at a wavelength of 430 nm after approx. 5 min.

Choose **a** cm^3 and **b** cm^3 test solution so that the absorbancy is in the upper quartile range of the calibration curve. The PO_4^{3-} content of the cigarette paper can be read from the calibration curve once the absorbancy has been corrected to 0,5 cm^3 test solution.

8. REPEATABILITY AND REPRODUCIBILITY

A collaborative test (8 laboratories / 6 replicates each) covering the range of 0,2-1,0% (m/m) phosphates in cigarette paper gave the following results:

r = repeatability

R = Reproducibility

see ISO 5725-2:1994

Mean PO ₄ ³⁻ (%) (m/m)	1,70	0,68	0,95
r (%)	0,06901	0,04056	0,02703
R (%)	0,196	0,102	0,129

9. TEST REPORT

The test report shall contain:

- date of test;
- material identification (brand name, supplier name, etc.);
- sampling procedure must be stated if different to ISO 186:1994;
- the paper conditioning parameters must be stated if different to ISO 187:1990;
- percent PO₄³⁻ in cigarette paper as mean value and standard deviation and number of replicate measurements;
- origin of paper (bobbin, spill etc.).

Note: when paper samples are taken from cigarettes (spills) the results may be influenced by external parameters (*e.g.* additives in the tobacco blend).