Cooperation Centre for Scientific Research Relative to Tobacco

Physical Test Methods Sub-Group

CORESTA Recommended Method No. 94

DETERMINATION OF CRUSH STRENGTH OF FLAVOUR CAPSULES FOR FILTERS – DEFINITIONS AND MEASUREMENT PRINCIPLES

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(August 2020)

1. SCOPE

This CORESTA Recommended Method specifies a physical test method for the determination of the crush strength of flavour capsules for filters.

2. NORMATIVE REFERENCES

All CORESTA Recommended Methods are subject to revision and parties to agreements based on these Recommended Methods are encouraged to investigate the possibility of applying the most recent editions of the methods indicated below.

ISO 3402
Tobacco and tobacco products – Atmosphere for conditioning and testing.

ISO 5893:2002
Rubber and plastics test equipment -- Tensile, flexural and compression types (constant rate of traverse) – Specification.

3. DEFINITIONS

3.1 Flavour Capsule

A capsule with flavour inside which is intended to be embedded in the filter of a tobacco product. The flavour is released, when the capsule is crushed by a compressive load.

3.2 Gauge Diameter

The initially measured diameter of the specimen.

NOTE: It is expressed in millimetres (mm).

3.3 Deformation

Change in diameter of the specimen, in the direction of the compressive load.

NOTE: It is expressed in millimetres (mm).

3.4 Crush Strength

The compressive load at which the specimen crushes in the test.

NOTE: It is expressed in Newton (N).

3.5 Crush Deformation

Change in diameter of the specimen, in the direction of the compressive load, at the time the specimen crushes in the test.

NOTE: It is expressed in millimetres (mm).

3.6 Crush Strain

Change in the diameter of the specimen, in the direction of the compressive load, relative to the gauge diameter.

NOTE: It is a dimensionless ratio.

3.7 Modulus of Elasticity

Ratio of crush strength to crush strain.

NOTE: It is expressed in Newton (N).

3.8 Load-Deformation Diagram

A diagram in which values of compressive load are plotted on the ordinate against corresponding values of deformation on the abscissa. Fig.1 is a typical Load-Deformation Diagram.

3.9 Test Speed

Rate of approach of the plates of the test apparatus during the test.

4. PRINCIPLE

The specimen is compressed between vertically approaching plates at constant speed until the specimen crushes. The load sustained by the specimen is measured during this process. The crush strength and the crush deformation are measured.

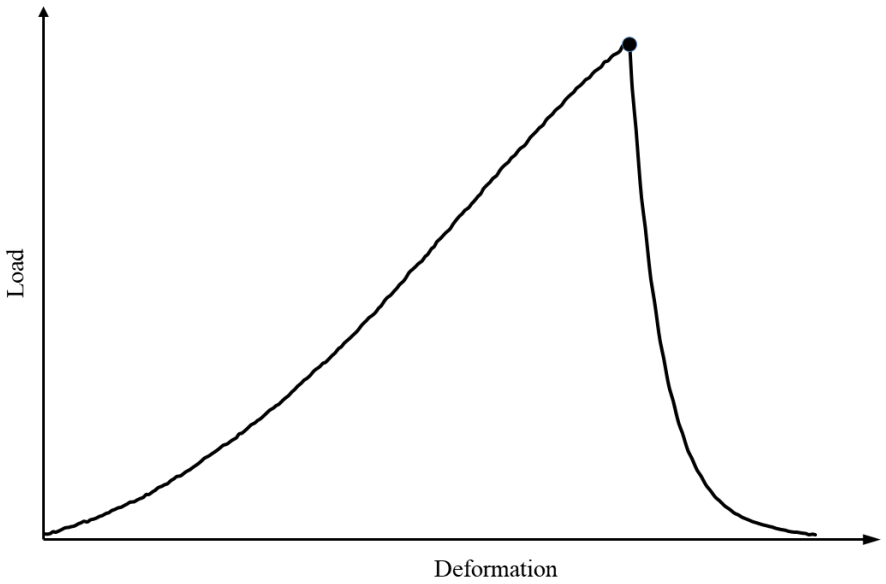

Fig.1 Typical Load-Deformation Diagram

5. SAMPLE CONDITIONING

5.1 Conditioning

Prior to each measurement, samples shall be conditioned in an atmosphere as specified in ISO 3402 (temperature (22 ±1) °C, relative humidity (60 ±3) %) for not less than 48 h.

5.2 Test conditions

Conduct measurements in the standard laboratory atmosphere as specified in ISO 3402 (temperature (22 ±2) °C, relative humidity (60 ±5) %).

6. APPARATUS

6.1 General

The apparatus used shall allow the vertical compression of the specimens with constant speed in order to determine the crush strength and crush deformation. The direction of compression shall be vertical.

6.2 Test Speeds

The apparatus shall be capable of maintaining the test speed to a tolerance of ±10 %.

6.3 Compression Plates

Hardened-steel compression plates shall be used to apply the compressive load to the specimen, so constructed that the load sustained by the specimen is transmitted through polished surfaces which are flat to within 0,025 mm, parallel to each other and perpendicular to the direction of the compressive load throughout the entire measurement process.

These plates shall be larger than the specimen to ensure that compressive load on the specimen is uniform. It is recommended that the diameter of the plates is not less than 16,0 mm.

6.4 Load Measurement System

A load measurement system capable of recording the compressive load sustained by the specimen. The system shall be essentially free of inertia lag at the specified test speed and shall indicate the value of the load with an accuracy of ±0,1 N or better.

6.5 Displacement Measurement System

A displacement measurement system capable of recording the deformation of the specimen during the testing. The system shall be essentially free of inertia lag at the specified test speed and shall indicate the value of the displacement with an accuracy of ±0,01 mm or better.

7. SAMPLING

Specimens shall be free of visible flaws or imperfections. Test at least thirty (30) specimens for each sample.

8. CALIBRATION OF APPARATUS

The measurement device shall be calibrated in accordance with the manufacturer's recommendations, ensuring the calibration procedure is done in accordance with ISO 5893:2002.

9. PROCEDURE

9.1 Conditioning of Specimens

Condition the specimens selected for the test as specified in 5.1.

9.2 Testing Atmosphere

Conduct the test in the standard atmosphere as specified in 5.2.

9.3 Calibration

Prior to use, set the measurement device to zero and calibrate the device using calibration standards and the calibration procedure in accordance with section 8.

9.4 Set-Up

Place the specimen between the compression plates ensuring that the specimen is in the centre of the plates.

9.5 Pre-Load

The specimen shall not be loaded prior to the test.

9.6 Test Speed

The test speed is (20 ±5) mm/min.

9.7 Test

Start the vertical motion of the plates to compress the specimen until it crushes.

9.8 Recording of Data

Record the load and the corresponding deformation. It is recommended to use an automatic recording system, which yields a complete load/deformation diagram for the test.

10. CALCULATION AND EXPRESSION OF RESULTS

10.1 General

Determine the deformation at the point where the load exceeds 0,1 N for the first time. This deformation is the initial deformation L_0.

Determine the load and the deformation at the point where the specimen crushes. This is the crush strength F_C and the maximum deformation L_C.

Record the distance of the plates at the point where the load exceeds 0,1 N for the first time. This distance is the gauge diameter D of the specimen.

10.2 Crush Deformation

Calculate the crush deformation, using the following equation:

$$\Delta L = L_C - L_0 \qquad (1)$$

where

ΔL is the crush deformation, expressed in millimetres;

L_C is the maximum deformation, expressed in millimetres;

L_0 is the initial deformation, expressed in millimetres.

10.3 Crush Strain

Calculate the crush strain, using the following equation:

$$\varepsilon = \frac{\Delta L}{D} \qquad (2)$$

where

ε is the crush strain, expressed as a dimensionless ratio;

ΔL is the crush deformation, expressed in millimetres;

D is the gauge diameter, expressed in millimetres.

10.4 Modulus of Elasticity

Calculate the modulus of elasticity, using the following equation:

$$E = \frac{F_C - F_0}{\varepsilon} \qquad (3)$$

where

E is the modulus of elasticity, expressed in Newton;

F_C is the crush strength, expressed in Newton;

F_0 is the initial load (0,1 N), expressed in Newton;

E is the crush strain, expressed as a dimensionless ratio.

10.5 Statistical parameters and significant figures

Calculate the arithmetic mean, the standard deviation, coefficient of variation, maximum and minimum of each set of test results.

Individual and mean values of crush strength and modulus of elasticity shall be expressed to one decimal place and their standard deviations to two decimal places. Individual and mean values of crush strain shall be expressed to two decimal places and the standard deviation to three decimal places.

11. PRECISION

An international collaborative study involving 9 laboratories was carried out in 2019/2020 to determine repeatability and reproducibility statistics of this test method. Five samples of flavour capsules were distributed to the laboratories and the laboratories were asked to perform 30 measurements on each sample to determine the crush strength, crush strain and modulus of elasticity. Tables 1a-c show the mean value (MV), and for repeatability and reproducibility the standard deviation (StD), the limit and the coefficient of variation (CoV) for a single measurement of crush strength, crush strain and modulus of elasticity.

Table 1a – Repeatability and reproducibility statistics for crush strength

	Crush Strength						
		Repeatability			Reproducibility		
ID	MV	StD	Limit	CoV	StD	Limit	CoV
	N	N	N	%	N	N	%
A	8,1	2,05	5,74	25,46	2,12	5,94	26,32
B	12,6	2,33	6,53	18,57	2,34	6,54	18,60
C	12,8	2,23	6,24	17,35	2,60	7,29	20,28
D	15,0	2,83	7,93	18,85	3,20	8,97	21,32
E	15,0	2,13	5,98	14,18	2,26	6,33	15,03

Table 1b – Repeatability and reproducibility statistics for crush strain

	Crush Strain						
		Repeatability			Reproducibility		
ID	MV	StD	Limit	CoV	StD	Limit	CoV
				%			%
A	0,28	0,075	0,209	26,737	0,091	0,254	32,510
B	0,37	0,055	0,155	14,796	0,068	0,190	18,156
C	0,39	0,041	0,115	10,393	0,051	0,144	13,023
D	0,37	0,047	0,131	12,701	0,052	0,147	14,303
E	0,43	0,038	0,106	8,815	0,047	0,132	10,915

Table 1c – Repeatability and reproducibility statistics for modulus of elasticity

	Modulus of elasticity						
		Repeatability			Reproducibility		
ID	MV	StD	Limit	CoV	StD	Limit	CoV
		N	N	%	N	N	%
A	29,4	2,26	6,33	7,67	4,12	11,53	13,99
B	33,8	2,42	6,78	7,17	4,27	11,97	12,66
C	32,5	3,05	8,55	9,40	5,11	14,31	15,74
D	40,5	3,19	8,93	7,89	5,47	15,33	13,53
E	34,9	2,64	7,40	7,57	3,76	10,53	10,77

For a determination of crush strength, crush strain and modulus of elasticity calculated from thirty individual measurements, the repeatability and reproducibility standard deviations (StD), the limits and the coefficients of variation (CoV) can be estimated from the values of Tables 1a-c and are provided in Tables 2a-c.

Table 2a – Repeatability and reproducibility statistics for crush strength

	Crush Strength						
		Repeatability			Reproducibility		
ID	MV	StD	Limit	CoV	StD	Limit	CoV
		N	N	%	N	N	%
A	8,1	0,37	1,05	4,65	0,39	1,08	4,81
B	12,6	0,43	1,19	3,39	0,43	1,19	3,40
C	12,8	0,41	1,14	3,17	0,47	1,33	3,70
D	15	0,52	1,45	3,44	0,58	1,64	3,89
E	15	0,39	1,09	2,59	0,41	1,16	2,74

Table 2b – Repeatability and reproducibility statistics for crush strain

	Crush Strain						
		Repeatability			Reproducibility		
ID	MV	StD	Limit	CoV	StD	Limit	CoV
				%			%
A	0,28	0,014	0,038	4,881	0,017	0,046	5,935
B	0,37	0,010	0,028	2,701	0,012	0,035	3,315
C	0,39	0,007	0,021	1,897	0,009	0,026	2,378
D	0,37	0,009	0,024	2,319	0,009	0,027	2,611
E	0,43	0,007	0,019	1,609	0,009	0,024	1,993

Table 2c – Repeatability and reproducibility statistics for modulus of elasticity

	Modulus of Elasticity						
		Repeatability			Reproducibility		
ID	MV	StD	Limit	CoV	StD	Limit	CoV
		N	N	%	N	N	%
A	29,4	0,41	1,16	1,40	0,75	2,11	2,55
B	33,8	0,44	1,24	1,31	0,78	2,19	2,31
C	32,5	0,56	1,56	1,72	0,93	2,61	2,87
D	40,5	0,58	1,63	1,44	1,00	2,80	2,47
E	34,9	0,48	1,35	1,38	0,69	1,92	1,97

It has to be noted that the testing according to this test method is destructive, thus the repeatability and reproducibility statistics contain the sample-to-sample variability, which is a substantial contribution to the overall variability. The values thus apply only to the specific sample material tested in this study and may be different for other sample materials.