TECHNICAL SPECIFICATION

ISO/TS 17996

IDF/RM 205

Second edition
2023-02

Cheese — Determination of rheological properties by uniaxial compression at constant displacement rate

Fromage — Détermination des propriétés rhéologiques par compression uniaxiale à vitesse constante de translation

Reference numbers
ISO/TS 17996:2023(E)
IDF/RM 205 :2023(E)

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Published in Switzerland

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Forewords

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This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*, and the International Dairy Federation (IDF). It is being published jointly by ISO and IDF.

This second edition cancels and replaces the first edition (ISO/TS 17996:2006 | IDF/RM 205:2006), which has been technically revised, with the following changes:

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

IDF (the International Dairy Federation) is a non-profit private sector organization representing the interests of various stakeholders in dairying at the global level. IDF members are organized in National Committees, which are national associations composed of representatives of dairy-related national interest groups including dairy farmers, dairy processing industry, dairy suppliers, academics and governments/food control authorities.

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This document was prepared by the IDF *Standing Committee on Analytical Methods for Processing Aids and Indicators* and ISO Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 5, *Milk and milk products*. It is being published jointly by ISO and IDF.

The work was carried out by the IDF/ISO Action Team on P18 of the *Standing Committee on Analytical Methods for Processing Aids and Indicators* under the aegis of its project leader Mr P. Watkinson (NZ).

Cheese — Determination of rheological properties by uniaxial compression at constant displacement rate

1 Scope

This document specifies a method for the determination of rheological properties by uniaxial compression at constant displacement rate in hard and semi-hard cheeses.

The method provides standard conditions for sampling and testing, for data representation and general principles of calculation.

NOTE Sampling can be difficult with some cheese varieties, e.g. caused by shortness, brittleness, stickiness and soft consistency. In these cases, reliable results cannot be achieved.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

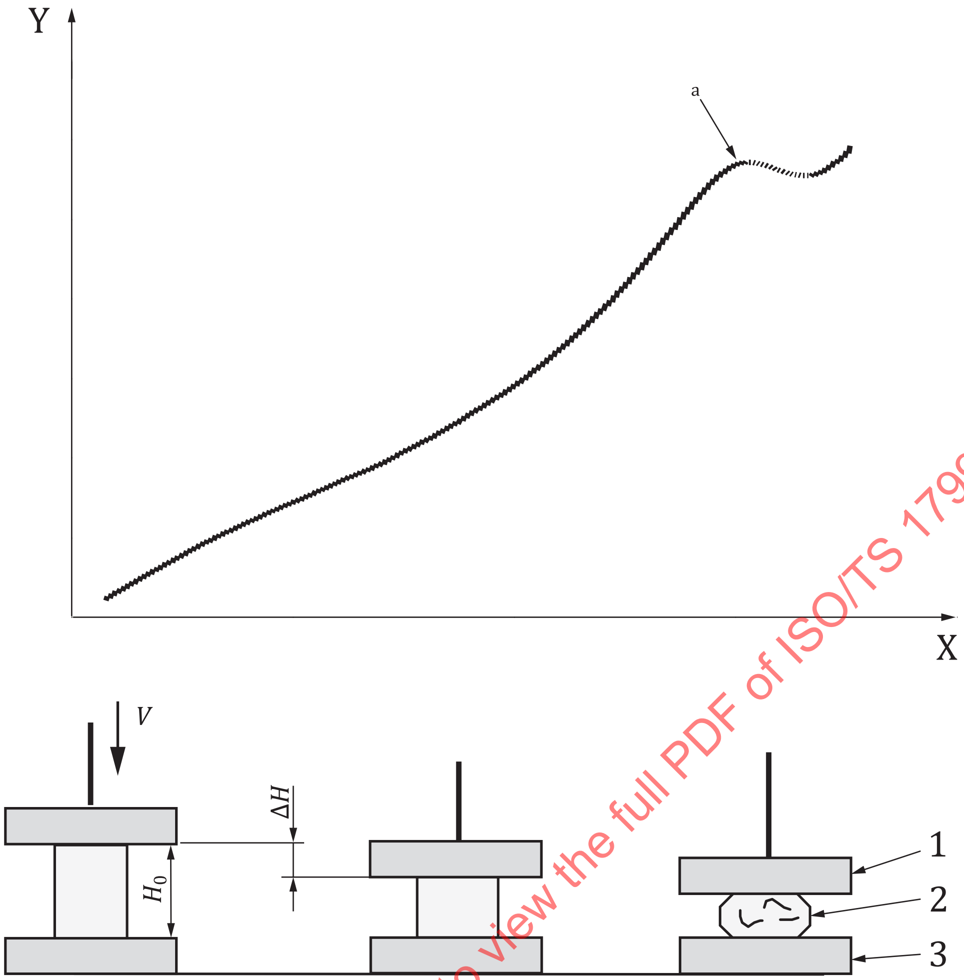
3.1
rheological properties
deformation under compression of the test sample

Note 1 to entry: In accordance with the procedure specified in this document.

4 Principle

A cylindrical test sample, of defined dimensions, is compressed at a constant crosshead speed with a compression tool up to a relative deformation sufficient to determine the apparent fracture point. The force, which is the resistance of the cheese sample during compression, is measured with a load cell. The displacement may be measured either from the position of the cross head or calculated from the elapsed time multiplied by the displacement rate.

A schematic representation of the principle of the test is given in Figure 1.



Key

X displacement

Y force

1 compression tool

2 sample

3 fixed base

V constant displacement rate of the compression tool

H_0 initial sample height

ΔH variation in sample height

[a] Apparent fracture point.

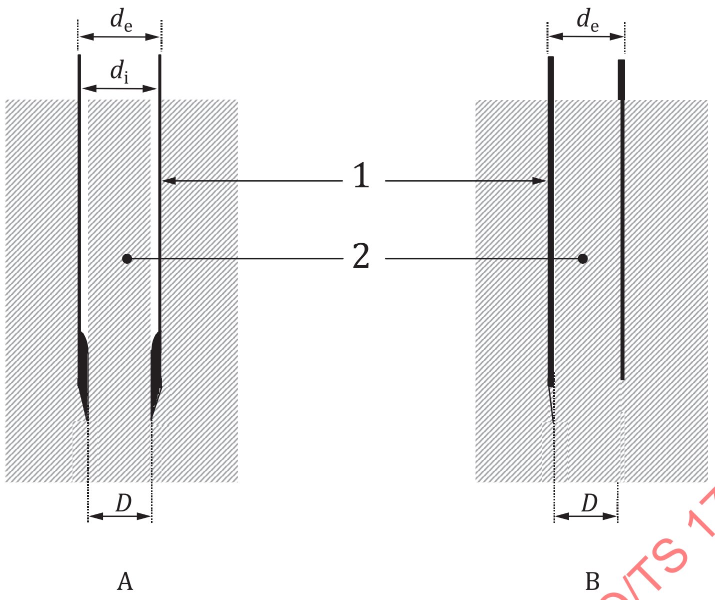
Figure 1 — Schematic principle of uniaxial compression at constant displacement rate

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Cork-borer, such as that shown in Figure 2 as an example.

It is recommended to mount the cork-borer on a drill-stand in order to drive it slowly and steadily through the test sample.



Key

1 cork borer

2 cheese plug

Example A: d_e external diameter (d_e = 25 mm)

d_i internal diameter (d_i = 23 mm)

D cutting head diameter (D = 20 mm)

Example B: d_e external diameter (d_e = 16,5 mm)

D cutting head diameter ($D = d_i$ is the internal diameter of 15,3 mm)

Figure 2 — Cork-borers for cutting cylindrical cheese plug

5.2 Parallel-wire cutting device, with a wire of diameter less than or equal to 0,4 mm and with a system to keep the two wires parallel to each other and perpendicular to the plug. It should also include a mechanically driven cutting system to cut the test sample to the required height.

5.3 Measuring cell, with a support and compression plate of the same stiff material, with smooth and parallel surfaces, e.g. stainless steel, aluminium or polytetrafluoroethylene (PTFE), of diameter larger (by 20 %) than that of the deformed test portion when at maximum compression. The load cell capacity shall have a reasonable relationship to the expected maximum force.

5.4 Compression instrument, providing compression functions typically consisting of two (or one) vertical columns on a platform and a crosshead connected perpendicular to these columns. This crosshead is driven vertically up and down by a motor. The load cell is typically directly connected to this crosshead and fixed to the compression tool (top plate) as shown in Figure 1. The fixed base in Figure 1 (bottom plate) is connected to the platform.

6 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 707 | IDF 50[1].

7 Procedure

7.1 Thermal equilibration of test samples

If the storage temperature of the loaf of cheese is above that of the measuring temperature, then the loaf of cheese shall be equilibrated at the measuring temperature for at least 50 h before further preparation of the test sample because of the slow crystallization of milk fat in the cheese.

If the storage temperature of the loaf of cheese is below that of the measuring temperature, before any preparation, store the loaf of cheese at the measuring temperature for at least 12 h. If there are specific difficulties that can occur during the sample preparation at the measuring temperature, then sample at the lower storage temperature and then equilibrate the test samples to the measurement temperature. In this case, the sample thermal equilibration time may be less than 12 h.

NOTE Examples of specific sampling difficulties are that the cheese is hard to cut, or a heated loaf of cheese changes the storage regime and therefore stops the use of the unsampled portions of the loaf of cheese for future measurements.

The following shall be avoided:

a) dehydration of the test sample during the period of thermal equilibration;

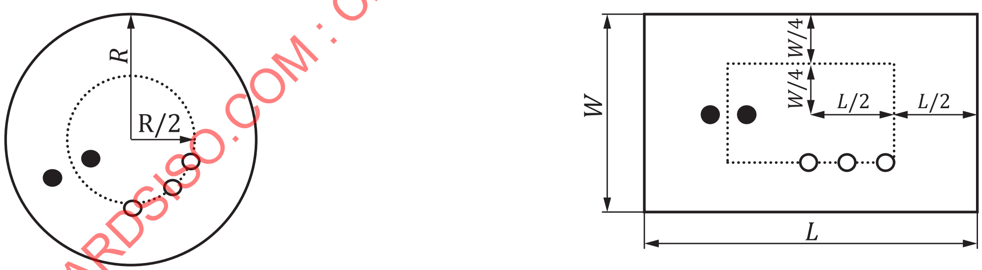
b) deformation of the test sample due to its own mass.

7.2 Test portion

7.2.1 Location

Take the test portion from the loaf of the cheese with a plug about half a radius, either along a circle of a cylindrical cheese, or along one side of a rectangular cheese (see Figure 3).

Cut the test portion in the plug in the area representing around half of the length (see Figure 4, plug A). If the height of cheese is sufficient, two portions can be taken as shown in Figure 4, plug B and plug C.

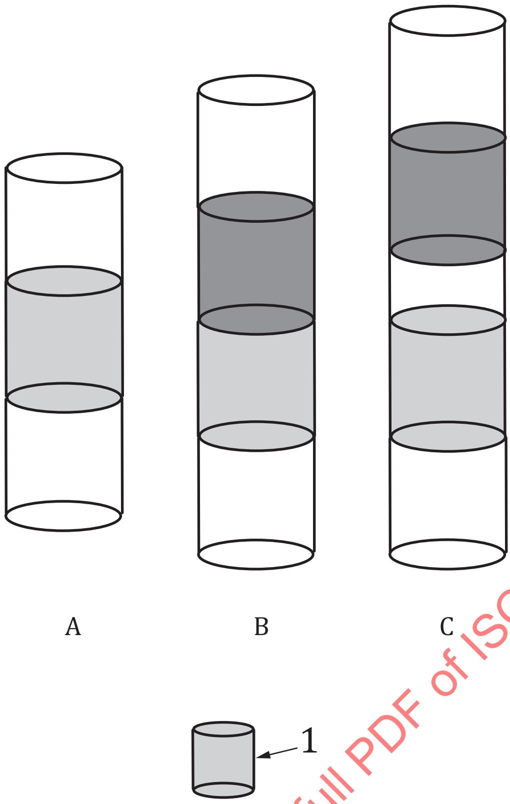


a) Cheese with cylindrical form b) Cheese with parallelepipedal form

Key

R radius
W width
L length
• plug in case 1
∘ plug in case 2

Figure 3 — Location of plug for cheese sampling



Key

1 sample

Figure 4 — Three types of sampling in plug

7.2.2 Direction

The standard direction for taking the test portion is parallel to the pressure axis in cheese making. See Annex B for non-standard sampling conditions.

7.2.3 Geometry

The shape of the test portion shall be a cylinder with initial height/diameter ratio (h_0/d_0) of between 1,1 and 1,5.

The initial height, h_0, of the test portion shall range from 12,5 mm to 25 mm. The diameter, d_0, for a given height follows the above-mentioned ratio.

7.2.4 Cutting

Remove the rind or the plastic cover. Take a test portion using a cork-borer (5.1) with shapes shown in Figure 2. For sticky cheeses, samples are easier to take with corer A than corer B. For cheese varieties showing shortness or brittleness, form B as shown in Figure 2 is more appropriate than form A. It is recommended to use a cork-borer mounted on a drill-stand in order to drive it slowly and steadily through the test sample.

If it is difficult to obtain a good cylindrical form, it is recommended to use mineral oil of low viscosity (e.g. Vaseline®[1]) oil) to lubricate the cork-borer. Do not test samples with cracks, holes or other visible defects.

1) Vaseline® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

Use a parallel-wire cutting device to cut the test sample to the required height. The wire diameter shall be less than or equal to 0,4 mm. It is essential to have a system that keeps the two wires parallel to each other and perpendicular to the plug. Preferably, use a mechanically driven cutting system. Taking these precautions into account reduces the lack in parallelism between the sample surface and the compression plate.

7.2.5 Delay

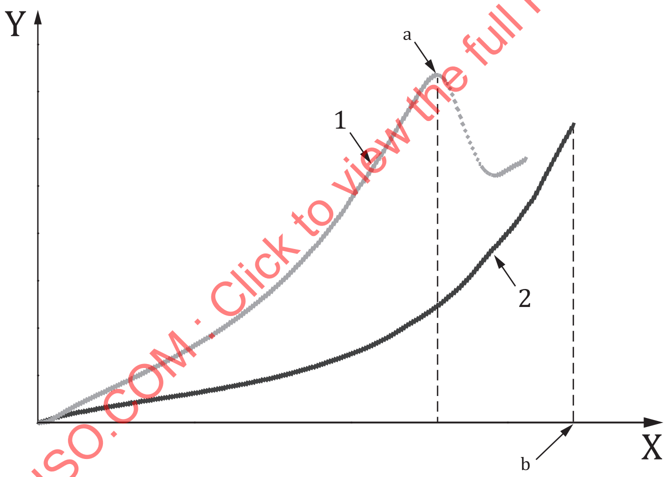
A delay between the taking of a test portion and its testing allows stress relaxation of the test portion. The recommended delay is between 10 min and 15 min. The upper limit is not strictly fixed but it should not exceed 2 h. This recommendation is not relevant when sampling is done at a lower temperature than the measuring temperature.

Store the test samples at the measuring temperature (see 7.3.5) and see Annex B for non-standard conditions. Store samples in a pill-box or wrapped in plastic film to avoid dehydration during the delay between sampling and testing.

7.3 Test conditions

7.3.1 Relative deformation

Perform the compression to just beyond the apparent fracture point (see Figure 5, curve 1) or to a predefined maximum deformation (see Figure 5, curve 2).



Key

X deformation

Y force

1 curve 1

2 curve 2

a Apparent fracture point.

b Maximum deformation.

Figure 5 — Examples of compression curves

7.3.2 Crosshead speed

The standard value of the crosshead speed or displacement rate is 50 mm/min (or 0,83 mm/s) for initial height 12,5 mm ≤ h_0 ≤ 25 mm.

7.3.3 Number of compression cycles

Perform one compression cycle.

7.3.4 Number of test portions

Measure at least four test portions, but preferably carry out more than this.

7.3.5 Measuring temperature

Measure at the standardized measuring temperature of 15 °C ± 1 °C.

NOTE Although the chosen test temperature of 15 °C is a good compromise for a single temperature, the challenge remains that many studies will use other temperatures for good reasons, as outlined in Annex A.

Follow Annex A for non-standard conditions.

7.3.6 Nature of the interface between test portion and plates

Use a low viscosity mineral oil as lubricant between the test portion and the plates. Apply the oil as a very thin layer on the plates.

8 Analysis of the compression curves

8.1 Data representation and calculation

8.1.1 Data representation

Raw data files contain data pairs (s_i, F_i) with displacement data, s_i, of the compression plate and force data, F_i, in units depending on the system. If the displacement of the plate is recorded right from the beginning of the test (i.e. before the plate is in contact with the sample), then compute the absolute deformation data, $|\Delta h_i|$, of the sample before any other calculation is performed. Let $|s_0|$ be the absolute displacement of the plate when the force becomes significantly different from zero (indicating the start of the compression of the sample). Then calculate the absolute sample deformation data, $|\Delta h_i|$, using Formula (1):

$$|\Delta h_i| = |s_i| - |s_0| \tag{1}$$

where

$|s_0|$ is the absolute displacement of the plate when the force becomes significantly different from zero;

$|s_\mathrm{i}|$ is the absolute displacement of the plate.

The correct sample deformation data, Δh_i, is then found using Formula (2):

$$\Delta h_i = -|\Delta h_i| \tag{2}$$

For further processing, it may be useful to remove data from the free precontact displacement of the compression plate.

If the measuring system automatically records the absolute deformation $|\Delta h|$ of the sample (starting with zero as soon as the contact force becomes significant), then $|s_i|$ is equal to $|\Delta h_i|$ and no correction has to be applied. However, the sign assignment $\Delta h_i = -|\Delta h_i|$ may still be necessary.

The deformation/force data (Δh, F) shall be transformed to the normalized measures strain and stress in order to be comparable. Graphical representations, numerical values and computed parameters of compression curves are given in terms of strain and stress. Both of the following representations to evaluate the curves are recommended options. Either one or both of these options may be used:

a) engineering stress, σ_u, versus Cauchy strain, ε_C;

b) corrected stress, σ_c, versus Hencky strain, ε_H.

Strain has no dimension; stress is given in pascals (Pa) or kilopascals (kPa).

Examples of compression curves from some cheese varieties are given in Annex B.

The symbol σ_u is also known as “uncorrected stress”; ε_C is the engineering strain or relative deformation. The terms “true stress” and “true strain” to denote corrected stress σ_c, and Hencky strain ε_H, respectively, are not recommended.

Stress correction is based on the cylindrical shape and volume constancy of the sample during the test, allowing the calculation of the cross-section, A_t, at each time point.

This changing cross-section, A_t, is calculated using Formula (3):

$$A_t = A(\Delta h) = \frac{A_0 \cdot h_0}{h(t)} \tag{3}$$

where

h_0 is the initial sample height;

A_0 is the initial cross-section;

$h(t)$ is the height at each time point is derived from h_0 and the deformation Δh, $h(t) = h_0 + \Delta h$. Under compression, Δh is a negative quantity ($\Delta h \leq 0$) because the deformation reduces the height of the sample, thus $h(t) = h_0 - |\Delta h|$ (see Reference [4]).

8.1.2 Calculation of stress and strain

Formulae (4), (5), (6) and (7) assume that $\Delta h \leq 0$.

The transformations of force into stress and of deformation into strain are applied to all data points (Δh, F).

$$\sigma_u = \frac{F_t}{A_0} \tag{4}$$

$$\varepsilon_C = \frac{\Delta h}{h_0} \tag{5}$$

$$\sigma_c = \frac{F_t}{A_t} = \frac{F_t}{A_0} \cdot \frac{h_t}{h_0} = \frac{F_t}{A_0} \cdot (1 + \varepsilon_C) = \sigma_u \cdot (1 + \varepsilon_C) \tag{6}$$

$$\varepsilon_H = \ln\left(\frac{h_t}{h_0}\right) = \ln\left(\frac{h_0 + \Delta h}{h_0}\right) = \ln(1 + \varepsilon_C) \tag{7}$$

NOTE 1 According to Formulae (5) and (6), compressive strain is a negative quantity (see Reference [4]). Although rheologically correct, it is not common practice to indicate the minus sign in graphical representations or in the computed parameters of the compression curves. The use of the sign can be useful if results from compression ($\varepsilon < 0$) and tension ($\varepsilon > 0$) need to be distinguished.

NOTE 2 The application of stress correction according to Formula (6) (see Reference [5]) induces a shift of the apparent fracture point towards lower absolute strain values (apart from decreasing the numerical stress values). This shift is a transformation property of the correction factor ($1 + \varepsilon_C$), applied to curves with local maxima (see Reference [7]).

8.2 Parameters characterizing the compression curves

8.2.1 General

Descriptive mechanical parameters of the compression curves are evaluated from stress/strain data calculated according to Formulae (4) to (7). The parameters characterize the first part of the curves and that part where fracture occurs. The apparent fracture work characterizes the curve up to the apparent fracture point.

The following four parameters are recommended to characterize the compression curves (see Annex B):

a) M_D, which is the modulus of deformability;

b) ε_f, which is the fracture strain (strain at the apparent fracture point);

c) σ_f, which is the fracture stress (stress at the apparent fracture point);

d) W_f, which is the fracture work (total deformation work up to the apparent fracture point, divided by the initial sample volume V; its value is equal to the area under the stress/strain curve from zero strain to strain at apparent fracture).

8.2.2 Modulus of deformability

The slope of the approximately linear part at small absolute strain values is an estimate of the apparent elastic modulus, also called "modulus of deformability", M_D.

A well-defined estimate of M_D is accessible by the maximum of the first derivative of the compression curve at $|\varepsilon| < 0{,}1$ using Formula (8):

$$M_D = \max\left(\frac{\partial\sigma}{|\partial\varepsilon|}\right)_{|\varepsilon|<0{,}1} \tag{8}$$

The calculation is applied to (ε_C, σ_U) and to (ε_H, σ_c) data.

The modulus of deformability, M_D, is given in pascals (Pa) or kilopascals (kPa).

The estimation of the modulus of deformability, M_D, according to Formula (8) is justified by the experimentally observed fact that there always exists a maximum of the first derivative at very low absolute strain. Although Formula (8) is recommended, other algorithms may be used to determine the modulus of deformability. The selection of a certain range $\Delta\varepsilon$ (with $|\varepsilon| < 0{,}1$) and the corresponding range $\Delta\sigma$ to calculate $M_D = |\Delta\sigma/\Delta\varepsilon|$, or the application of linear regression over a certain number of (ε,σ) pairs is also possible to estimate M_D.

Compression curves without characteristic features (i.e. monotonously increasing stress, no fracture point, no inflection point) can occur in very young hard and semi-hard cheeses. Though this case is outside the scope of this document, and the compression test not well adapted to study this behaviour, the curves may be evaluated by determining several data points (ε,σ) at sensibly chosen strain values. The estimation of the modulus of deformability, M_D, according to Formula (8) is still possible. In fact, the maximum of the first derivative at low strain is the only feature of such curves.

8.2.3 Apparent fracture point

8.2.3.1 General

The local maximum of the compression curve is defined as the apparent fracture point. Therefore, the fracture strain ε_f and fracture stress σ_f are also "apparent" quantities. Both parameters are determined from (ε_C, σ_U), and from (ε_H, σ_c) data.

8.2.3.2 Determination of curve maximum

The determination of curve maximum is simple if the curve shows one local maximum that is clearly identifiable as the apparent fracture point. In that case, calculate the maximum stress value, σ_f, using Formula (9):

$$\sigma_f = \max(\sigma_i)_{1 \le i \le n} \tag{9}$$

where n is the number of data points describing the compression curve to just beyond the apparent fracture point (see 8.2.3).

Calculate the strain value, ε_f, which corresponds to the maximum stress of the curve using Formula (10):

$$\varepsilon_f = \varepsilon(\sigma_f) \tag{10}$$

8.2.3.3 Determination from an inflection point of the curve

If there is no maximum assignable to the apparent fracture point but only a well-established inflection point indicating fracture, the coordinates of this point are taken as an approximation of the fracture stress σ_f and fracture strain ε_f. The determination of this inflection point is best achieved via the second derivative of the compression curve (the first derivative of the curve shows a local maximum and the second derivative is zero at this inflection point). If it is the only inflection point of the curve, the fracture strain ε_f may be found using Formula (11), which is the strain value ε_i where the second derivative is zero:

$$\varepsilon_f = \varepsilon_i \text{ with } \frac{\partial^2 \sigma(\varepsilon_i)}{\partial \varepsilon_i{}^2} = 0 \tag{11}$$

Accordingly, Formula (12) holds for the fracture stress:

$$\sigma_f = \sigma(\varepsilon_f) \tag{12}$$

Fracture strain has no dimension; fracture stress is given in pascals (Pa) or kilopascals (kPa).

The determination of the fracture parameters σ_f and fracture strain ε_f from the local maximum of the curve according to Formulae (9) and (10) is easily programmable. In the case of approximating the apparent fracture point from the coordinates of an inflection point according to Formulae (11) and (12), the algorithm used to find ε_f shall be such that it finds the correct value (compression curves can have several inflection points). In practice, it may be sufficient to define the direction of the searching procedure from high to low absolute strain values. It is recommended to check the numerical values found in this way by inspecting the plots.

NOTE It is not possible to calculate ε_f (Hencky) and σ_f (corrected) from ε_f (Cauchy) and σ_f (uncorrected) by applying Formulae (7) and (6), respectively.[7] All four results are correct only if the algorithms are independently applied to both data representations. The reason is the shift of the maximum mentioned in 8.1.2.

8.2.4 Apparent fracture work

The apparent fracture work, W_f, is defined as the total work of deformation applied during the test to the sample in the strain interval $\{0, \varepsilon_f\}$, divided by the initial sample volume V_0. This value is equal to the area under the stress/strain curve from zero strain to strain at apparent fracture.

Calculate the apparent fracture work, W_f, according to Formula (13), which applies to both representations but is not valid in mixed representations such as (ε_C, σ_c) or (ε_H, σ_u). W_f is expressed in units of J/m^3 or kJ/m^3, depending on the units of σ (ε) used in Formula (13) (in Pa or kPa, respectively):

$$W_f = \left| \int_0^{\varepsilon_f} \sigma(\varepsilon) \, d\varepsilon \right| \tag{13}$$

NOTE The numerical integration can be performed using the discrete data points, replacing the integral sign by the appropriate sum over the work per volume increments $|\Delta\varepsilon\sigma(\varepsilon)|$. The approximation has been proven to give correct results.

$$W_f = \sum_{i=1}^{i(\varepsilon_f)-1} \left| \varepsilon_{i+1} - \varepsilon_i \right| \cdot \frac{\sigma_{i+1} - \sigma_i}{2} \tag{14}$$

8.3 Expression of results

Express the test results for the modulus of deformability (unit: kPa) to one decimal place.

9 Precision

9.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are given in Annex C. However, this test did not fulfil the requirements of an interlaboratory test in accordance with ISO 5725-1[2] and ISO 5725-2[3]. Thus, this document is a Technical Specification rather than an International Standard.

The values for repeatability and reproducibility limit are not necessarily applicable to ranges and matrices other than those given.

9.2 Repeatability

The absolute difference between two individual single test results, obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than the values given in Table 1.

Table 1 — Repeatability values

Parameter	a)	b)
Modulus of deformability (kPa)	324,7	278,7
Fracture strain	0,04	0,065
Fracture stress (kPa)	50,76	30,85
Work at fracture (kJ/m^3)	13,60	9,52

a Engineering stress versus Cauchy strain.

b Corrected stress versus Hencky strain.

9.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than the given in Table 2.

Table 2 — Repeatability values

Parameter	a)	b)
Modulus of deformability (kPa)	343,6	457,9
Fracture strain	0,36	0,460
Fracture stress (kPa)	135,62	78,88
Work at fracture (kJ/m^3)	32,03	25,27

[a] Engineering stress versus Cauchy strain.

[b] Corrected stress versus Hencky strain.

10 Test report

The test report shall specify:

a) all information necessary for the complete identification of the sample;

b) the sampling method used, if known;

c) the test method used, together with reference to this document, i.e. ISO/TS 17996 | IDF/RM 205;

d) all other operating details not specified in this document, or regarded as optional, together with details of any incidents which can influence the test result(s);

e) the test result(s) obtained or, if the repeatability has been checked, the final quoted result obtained;

f) the date of the test.

Annex A
(normative)

Non-standard sample conditions

A.1 Direction of sampling

Although it is recommended to take a test portion parallel to the pressure axis, for special purposes a test portion at right angles to that may be of interest.

A.2 Geometry of the portion

A cylindrical form of the test portion is preferred for the following reasons:

a) a regular shape is easiest;

b) there is homogeneity of stress distribution;

c) the symmetry makes mathematical calculations easier.

If h_0 is between 12,5 mm and 25 mm, a constant displacement rate of 50 mm/min means an initial strain rate between 0,067 s^{-1} and 0,033 s^{-1} (see Table A.1). If h_0 is outside the range of 12,5 mm to 25 mm, adjust the displacement rate, v, so as to reach an initial strain rate equivalent to the reference value obtained with h_0 = 15 mm and v = 50 mm/min by using the following relation: $v = 0{,}56\ h_0$.

Table A.1 — Initial strain rate values corresponding to different initial test portion heights

h_0 (mm)	**Initial strain rate**[a] (s^{-1})
12,5	0,067
15	0,056
20	0,042
25	0,033

[a] Initial Cauchy strain rate ($\varepsilon_C = v/h_0$) and Hencky strain rate [$\varepsilon_H = v/(h_0 - vt)$] at $t = 0$ are equal.

A.3 Testing temperature

It is difficult to fix one measuring temperature for several reasons. Often the lack of a temperature-controlled room or system limits the choice in practice. When rheological measurements are performed to study changes during maturation, the temperature of the maturing rooms depends on the cheese variety and can change during ripening. In that case, the maturing temperature preferably determines the choice of the measuring temperature. In other cases, rheological measurements are performed to study relationships with sensory analysis, and then a measuring temperature close to the temperature of the products during sensory tests is preferred. Experiences in sensory tests have shown that a temperature of 15 °C to 16 °C is quite appropriate.[6] Consequently, the optimal measuring temperature strongly depends on the aim of the studies.

A.4 Load cell

When these results need to be reported, it is recommended to add explicitly the following to the test report (see Clause 9):

a) the lowest measurable force;

b) the resolution of the load cell, i.e. the smallest difference of force that can be measured significantly.