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Standard Safety Specification for Liquid Laundry Packets[1]

This standard is issued under the fixed designation F3159; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Editorially corrected 5.3.3.2, 6.1, and 6.2 in December 2015.

INTRODUCTION

In November 2012 the U.S. Consumer Product Safety Commission (CPSC) issued a Safety Alert to inform parents and caregivers that Liquid Laundry Detergent Packets need to be kept away from children as those who are exposed to packet contents are at risk of serious injury and even death due to the highly concentrated nature of the product. Children who have accidently ingested Liquid Laundry Detergent Packets have experienced a range of injuries including loss of consciousness, respiratory distress, vomiting, coughing, choking and drowsiness, and in cases where there has been contact with the eyes, painful irritation of the eyes and corneal burns have occurred. In addition, death has been reported to occur following ingestion of Liquid Laundry Detergent Packets, including in one child.

1. Scope

1.1 This specification provides requirements for household Liquid Laundry Detergent Packet safety to help reduce unintentional exposures to the contents of the packets, especially to children.

1.2 This standard applies exclusively to household Liquid Laundry Detergent Packets. "Liquid Laundry Detergent Packets" are single-use laundry detergent products that contain a liquid detergent enclosed in a water soluble outer layer ("pouch film"). This includes laundry detergent packets in soluble film that contain liquid only (that is, all liquid), as well as those that contain both liquid and non-liquid components.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with Liquid Laundry Detergent Packet use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. It is the responsibility of the user of the product to follow the warning statements and use the product appropriately.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3475 Classification of Child-Resistant Packages

D4332 Practice for Conditioning Containers, Packages, or Packaging Components for Testing

D4359 Test Method for Determining Whether a Material Is a Liquid or a Solid

2.2 *ANSI Standard:*[3]

ANSI Z535.4 Safety Color Code—Environmental Facility Safety Signs—Criteria for Safety Symbols—Product Safety Sign and Labels and Accident Prevention Tags

3. Terminology

3.1 *Definitions:*

3.1.1 *liquid, n*—a substance or mixture which: *(1)* at 50°C has a vapour pressure of not more than 300 kPa (3 bar), *(2)* which is not completely gaseous at 20°C and at a standard pressure of 101.3 kPa, and *(3)* which has a melting point or initial melting point of 20°C or less at a standard pressure of 101.3 kPa.

[1] This specification is under the jurisdiction of ASTM Committee F15 on Consumer Products and is the direct responsibility of Subcommittee F15.71 on Liquid Laundry Packets.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.1.1.1 *Discussion*—A viscous substance or mixture for which a specific melting point cannot be determined shall be subjected to the Test Method D4359-90 test; or to the test for determining fluidity (penetrometer test) prescribed in section 2.3.4 of Annex A of the European Agreement concerning the International Carriage of Dangerous Goods by Road (ADR).

3.1.2 *liquid laundry detergent packets, n*—individual packets that contain liquid laundry detergent and are intended to dissolve when used as intended.

3.1.3 *pouch film, n*—the water-soluble outer layer of a Liquid Laundry Detergent Packet that contains laundry detergent or other liquid ingredients, or both, and is designed to dissolve when used as intended.

4. Liquid Laundry Detergent Packet Requirements

4.1 The Liquid Laundry Detergent Packet must meet the requirements set forth in European Commission Regulation (EU) No 1272/2008, Annex II, Part 3, Section 3.3.3, and Sections 4 (Aversive Agent in the Soluble Film) and 5 (Capsule Integrity) of the accompanying AISE Liquid Laundry Detergent Capsules Guidelines on CLP Implementation[4], including as may be amended. For reference, EU No 1272/2008, Annex II, Part 3, Section 3.3.3, is attached as Annex A5, and Sections 4 and 5 of the AISE Liquid Laundry Detergent Capsules Guidelines on CLP Implementation are attached as Annex A7.

4.2 For the avoidance of doubt, European Commission Regulation (EU) No 1272/2008, Annex II, Part 3, Section 3.3.3 (i) does not specify any particular manner in which the soluble packaging containing the agent must have the aversive agent added to the soluble packaging. A company may choose, by way of non-limiting examples, to introduce the aversive agent to the soluble packaging by admixing it into a slurry that is subsequently formed or cast into a film and/or by coating it onto a previously formed film. It is up to each company to select the aversive agent and technology they deem appropriate for their products and effective for meeting the criteria of being safe and eliciting oral repulsive behavior within a maximum time of 6 s as provided by and in accordance with the EU Regulation.

5. Packaging Requirements

5.1 Liquid Laundry Detergent Packets shall be contained in opaque packaging or packaging that employs any equivalent measure intended to mask the visibility of the individual Liquid Laundry Detergent Packets (“Package” or “Packaging”). The package must not be labeled with graphics that make the opaque package appear transparent or translucent.

5.2 Packaging described in this Voluntary standard is packaging that is designed or constructed to be difficult for children to access Liquid Laundry Detergent Packets. To comply with this standard, the package shall have the characteristics of at least one of the following six options outlined below in 5.3.1. In addition to meeting with at least one of the six options, a package must also:

5.2.1 Comply with this standard through the full life cycle of product package.

5.2.2 The package must meet the option standard for which the package is designed while also accounting for any other way the package could conceivably be opened for example by twisting, pulling, or use of singular force.

5.3 *Options for Packaging:*

5.3.1 A package that meets the performance requirements of 16 CFR Part 1700, section 1700.15 and testing requirements of 16 CFR Part 1700, section 1700.20.

5.3.2 An individually-wrapped package that contains no more than one packet and incorporates either:

5.3.2.1 A hidden tab or notch or other means of opening that is only exposed after the package has been folded or manipulated in an instructed manner, or

5.3.2.2 A feature described in Classification D3475-14, Type IV Non-reclosable packaging-flexible and Type V Unit non-reclosable packaging-rigid.

5.3.3 A package that requires manipulative skill or dexterity to open, including, but not limited to:

5.3.3.1 A package with two or more closure mechanisms that are interdependent, so that the package cannot be fully opened without releasing at least two of the closure mechanisms.

5.3.3.2 A double-action release mechanism, defined as either:

(1) A mechanism requiring two consecutive motions, the first of which must be maintained (and which may include the act of physically holding or stabilizing the package) while the second is carried out in order to fully open the package, or

(2) Two separate and independent motions that must be activated or occur simultaneously to fully open the package.

NOTE 1—A simple zipper closure (pull to open or simple slider) would not meet the requirements of either 5.3.3.2*(1)* or *(2)*.

5.3.3.3 A release mechanism or system of mechanisms which requires two independent release mechanisms to be performed consecutively in order to fully open the package.

5.3.3.4 The package must be designed so that the user is able to close the package and re-engage the release mechanism(s) in a manner that requires only one re-engagement action on the part of the user.

5.3.4 A package closure that meets the requirements set forth in European Commission Regulation (EU) No 1272/2008, Annex II, Part 3, Section 3.3.2 (iv), and Section 3.3 (Outer Packaging: Closures) of the accompanying AISE Liquid Laundry Detergent Capsules Guidelines on CLP Implementation, as may be amended. For reference, EU No 1272/2008, Annex II, Part 3, Section 3.3.2 (iv), is attached as Annex A4, and Section 3.3 of the AISE Liquid Laundry Detergent Capsules Guidelines on CLP Implementation is attached as Annex A6.

5.3.5 A package that requires the intellectual skill or cognitive ability of a child at least 6 years of age to open, that meets all of the criteria set forth in 5.3.5.1 – 5.3.5.3, or a reasonable equivalent of 5.3.5.1 – 5.3.5.3, and meets the requirements of 5.3.5.4:

[4] A.I.S.E., Liquid Laundry Detergent Capsules Guidelines On CLP Implementation, Version 1.0, 27 February 2015.

5.3.5.1 Identification of a non-obvious opening method that requires cognitive understanding of a manipulative concept.

5.3.5.2 The mechanical means that secures the package is obscured from view and not readily apparent when handling the package.

5.3.5.3 Requires manipulation of a visual or tactile feature in a way that is non-obvious unless the user understands the manipulative concept.

5.3.5.4 The package must be designed so that the user is able to close the package and re-engage the release mechanism(s) in a manner that requires only one re-engagement action on the part of the user.

5.3.6 A package that, in order to be opened, requires either:

(1) An opening force greater than that which a child is capable of generating while not being greater than a senior adult is capable of generating, or

(2) Hand anthropometric characteristics greater than those of an average-sized child.

5.3.6.1 The ages and distribution of children to be tested for purposes of establishing the opening force strength or hand anthropometric characteristics must meet the requirements set forth in 16 CFR 1700.20(a)(2)(i).

5.3.6.2 The opening force strength or hand anthropometric characteristics shall be set at the 95th percentile for children, and the 5th percentile for senior adults.

5.3.6.3 Research must demonstrate that the opening force strength or hand anthropometric characteristics do not exceed those of senior adults between the ages of 50 and 70 years to access the package, as tested pursuant to the requirements set forth in 16 CFR 1700.20(a)(3).

5.3.6.4 Research that establishes opening strength or hand anthropometric characteristics as set forth above must be conducted by an independent third party, and the results published in a peer-reviewed journal.

5.3.6.5 A manufacturer must demonstrate through testing that the opening force required or hand anthropometric characteristics for its packaging design are within the limits of the data.

6. Labels

6.1 Each package shall be labeled with warning statements. The warning statements shall be:

6.1.1 In contrasting color(s), permanent, conspicuous, and in sans serif style font;

6.1.2 Distinctively separated from any other wording or graphics, in a "quiet zone" (that is, placed on a single-color, contrasting background); and

6.1.3 Located on the product in a prominent location so they are visible to the consumer.

6.2 The following statements shall appear on the front panel/principal display panel of the package:

WARNING:

Harmful if put in mouth or swallowed. Eye irritant.

Packets can burst if children put them in mouth or play with them.

See warning on [back/side] label.

Keep out of reach of children.

6.2.1 See Annex A1 for an example of an FHSA-compliant layout.

6.3 The Safety Alert Symbol and text of the precautionary statements shall be laid out on the back or side panel of the secondary container as set forth in ANSI Z535-4 (2011) Figures 3 through 12 and Figures B26 through B28 or in a substantially similar format, except that neither the borders nor boxes depicted in those figures are required. The precautionary statements shall include the following:

6.3.1 The Safety Alert Symbol, as found in ANSI Z535-4, Fig. 1 (D) or (E).

6.3.2 Annex A2 includes additional safety symbols to be used with the warning statements. Each package must feature at least one pair (that is, one "keep out of reach of children" symbol and one "keep contents out of eyes" symbol).

6.4 The following statements shall appear on the back or side panel of the secondary container:

WARNING:

Concentrated detergent packets can burst if children put them in mouth or play with them. The liquid inside is harmful if put in mouth, swallowed, or in eyes.

Keep packets out of reach of children.

- Store container where children cannot reach or climb to it, out of sight and in a secure place.
- Keep container fully closed.
- Never leave any packets out of container.
- DO NOT let children handle packets, even if supervised.

Avoid breaking packets.

- Do not handle packets with wet or moist hands. Do not expose packets to moisture.

FIG. 1 Safety Alert Symbol

• Do not cut or puncture packets. If packets stick together, do not try to separate them.

Call poison control center immediately if detergent gets in mouth or eye or on skin. Immediately and thoroughly rinse eye or skin with water for 15 min.

6.5 All individually-wrapped sample packages shall contain no more than one packet. Each individually-wrapped sample package shall include the following statements:

WARNING:

Concentrated detergent packets can burst if children put them in mouth or play with them. The liquid inside is harmful if put in mouth, swallowed, or in eyes.

Keep packet out of reach of children.

• Store where children cannot reach or climb to it, out of sight and in a secure place.

• DO NOT let children handle packet, even if supervised.

• Use packet immediately after opening.

Avoid breaking packet.

• Do not handle packet with wet or moist hands. Do not expose packet to moisture.

• Do not cut or puncture packet.

Call poison control center immediately if detergent gets in mouth or eye or on skin. Immediately and thoroughly rinse eye or skin with water for at least 15 min.

6.6 Each individually-wrapped packet that is contained in a larger outer package that contains multiple individually-wrapped packets shall include at least one pair (that is, one "keep out of reach of children" symbol and one "keep contents out of eyes" symbol) of the additional safety symbols from Annex A2.

6.7 The language listed above and icons shown in Annex A2 may be modified as necessary to ensure compliance with local regulatory requirements, or for translation purposes. Additional warnings or cautionary statements or, if appropriate, alternate first aid instructions may also be included on the label, depending on the formula, packaging used and other considerations. Furthermore, and for the avoidance of doubt, other words, such as "pac" or "pack" or a trademarked name for the product, may be substituted for "packets" in these statements in order to allow for consistent terminology on each product's package.

7. Keywords

7.1 child deterrent; container; detergent; ingestion; laundry packet

ANNEXES

(Mandatory Information)

A1. FHSA-COMPLIANT PRINCIPAL DISPLAY PANEL (FRONT PANEL)

WARNING: HARMFUL IF PUT IN MOUTH OR SWALLOWED. EYE IRRITANT. Packets can burst if children put them in mouth or play with them. See warning on [back/side] label.

Keep out of reach of children.

A2. ICON AND ALERT SYMBOL EXAMPLES

A2.1 See Fig. A2.1.

NOTE 1—If words are not included within the prohibition surround shape, use standard prohibition circle (rather than the version that is widened to accommodate words, as seen in the first example).

FIG. A2.1 Icon and Alert Symbol Examples

A3. OTHER INFORMATION SOURCES RELATING TO THE SAFETY OF LIQUID LAUNDRY DETERGENT PACKETS

ACCC, http://www.productsafety.gov.au/content/index.phtml/itemId/999447 & http://www.productsafety.gov.au/content/index.phtml/itemId/998653/fromItemId/999447

Accord, http://www.accord.asn.au/public_information_submission/children_and_safe_storage

ACI (USA), http://www.cleaninginstitute.org/clean_living/singleload_liquid_laundry_packets.aspx

AISE (Europe), http://www.aise.eu/go.php?pid=44122&topics=1

CPSC (USA), http://www.cpsc.gov//PageFiles/132488/390%20Laundry%20Packets.pdf

A4. EUROPEAN COMMISSION REGULATION (EU) NO 1272/2008, ANNEX II, PART 3, SECTION 3.3.2(iv)

A4.1 Without prejudice to the requirements of section 3.1, be fitted with a closure that: (a) impedes the ability of young children to open the packaging by requiring coordinated action of both hands with a strength that makes it difficult for young children to open it; (b) maintains its functionality under conditions of repeated opening and closing for the entire life span of the outer packaging.

A5. EUROPEAN COMMISSION REGULATION (EU) NO 1272/2008, ANNEX II, PART 3, SECTION 3.3.3

A5.1 The soluble packaging shall:

A5.1.1 Contain an aversive agent in a concentration which is safe, and which elicits oral repulsive behaviour within a maximum time of 6 s, in case of accidental oral exposure;

A5.1.2 Retain its liquid content for at least 30 s when the soluble packaging is placed in water at 20°C;

A5.1.3 Resist mechanical compressive strength of at least 300 N under standard test conditions.

A6. A.I.S.E. LIQUID LAUNDRY DETERGENT CAPSULES GUIDELINES ON CLP IMPLEMENTATION (SECTION 3.3)

A6.1 Closures

A6.1.1 The closure of the LLDC outer packaging must meet two main requirements that need to be balanced:

A6.1.1.1 impede young children from opening the packaging and

A6.1.1.2 for adults, allow easy regular opening and reclosing after use.

A6.1.2 These functionalities must be maintained during the packaging life span.

A6.1.3 In addition, the pack (that is, the ‘outer packaging’ in the Soluble Packaging Regulation) should be self-standing and should remain so throughout the life span of the pack.

A6.1.4 With regard to closure design, the Soluble Packaging Regulation refers qualitatively to two elements: ‘requiring coordinated action of both hands’ and ‘a strength’ for opening.

A6.1.5 These requirements apply ‘without prejudice to the requirements of section 3.1 [of Annex II to CLP]’ which prescribe child-resistant fastenings for specific mixture classifications (such as skin corrosive products). A.I.S.E.’s understanding is that the closure requirements for Soluble Packaging are different from child-resistant fastenings in section 3.1 and apply independently, without conflict. So section 3.1 of Annex II continues to apply for certain mixture classifications and, in addition, the new section 3.3 applies to LLDCs regardless of their classification.

A6.1.6 No performance standards exist today for ‘child-impeding closures’ that are not fully ‘child-resistant’ (in the meaning of the ISO 8371 standard). Our industry is committed to work on the development of a performance standard to assess the ‘child-impeding’ function of packaging, taking into account that ‘coordinated action of both hands’ is required.

A6.1.7 It should be noted that it would require at least two years to shelf new packaging designs in all markets.

A6.1.8 In the meantime, A.I.S.E. suggests the following:

A6.1.8.1 ‘coordinated action of both hands’ for opening: in the lack of clear design description in the legal text, it is up to each company to assess the design against compliance with this general requirement. It builds on the fact that the key differentiator between adults and children is mental capacity, logic

and dexterity. Coordination may include the required use of hands to secure a pack to enable the opening of a closure system (for example, stand-up pouches).

A6.1.8.2 'with a strength' for opening: is to be seen in the context of the target age group, namely children below the age of 6 years. No strength value is specified in the legal text but it should be sufficient so that the closure cannot be opened unintentionally (for example, by simply touching the outer packaging). Again, it should be borne in mind that the key differentiator between adults and children is dexterity and logic rather than strength.

A6.1.8.3 'easily reclosable': the outer packaging closure must be able to be closed by adults in a single action, such as but not limited to, one clip to be pushed, a gentle pressure on the lid to lock, one zipper to be activated.

A6.1.8.4 'maintains its functionality under conditions of repeated opening and closing for the entire life span': the closure system must meet the above criteria on opening and reclosing for the designed life of the packaging, which corresponds to at least the number of capsules/unit doses in the outer packaging.

A7. A.I.S.E. LIQUID LAUNDRY DETERGENT CAPSULES GUIDELINES ON CLP IMPLEMENTATION (SECTIONS 4 AND 5)

A7.1 Aversive Agent in the Soluble Film

A7.1.1 According to the Soluble Packaging Regulation, the soluble packaging (that is, the capsule wall) must **contain an aversive agent** in a concentration which is safe, and which elicits oral repulsive behavior within a **maximum time of 6 s**, in case of accidental oral exposure.

A7.1.2 This measure is intended to further reduce the chance of ingestion of the liquid content in case a child left unattended has managed to gain access to a capsule and places it in his/her mouth.

A7.1.3 A.I.S.E. has developed and evaluated a **protocol to determine effective levels of aversive agent** contained in soluble packaging, that is, in the soluble film. The resulting study protocol is provided in Annex A8.

A7.1.4 The objectives of this work were:

A7.1.4.1 to develop a *method for measuring the oral rejection time*, as a function of the level of aversive agent in the film;

A7.1.4.2 to prove the concept of effectiveness testing (at different concentrations of aversive agent), in other words to establish a 'benchmark test'.

A7.1.5 One grade of film and one particular aversive agent were selected for the study.

A7.1.6 The A.I.S.E. study has shown that, for the particular aversive agent and film selected, it was possible to determine a level of aversive agent sufficient to elicit a median oral rejection in less than 6 s. Above this concentration, the 'dose-response' curve was flat, that is, higher levels of aversive agent were not found to lead to lower rejection times. A summary of the study findings is provided in Annex A9.

A7.1.7 For ethical reasons, the study was run on young adults instead of children. This is a conservative approach, because a child's palate is much more sensitive than that of adults. Infants have around 30 000 taste buds spread throughout their mouths. By the time adulthood is reached, only about a third of these remain, mostly on the tongue. The decreasing sensitivity to bitterness with age was demonstrated by Mennella et al. (2005)[5]. Consequently, it is reasonable to assume that the observed oral rejection times with young adults are similar to or higher than what may be expected with young children.

A7.1.8 It is important to note that it is up to each company to demonstrate effectiveness of the aversive agent chosen to their own situation (soluble film/agents) at *design* stage. This is because:

A7.1.8.1 different aversive agents may lead to different human responses and

A7.1.8.2 the effective concentration of aversive agent may be affected by the polymer chemical composition, presence of other chemicals in the film, etc.

A7.1.9 It is also up to each company to select the aversive agent they deem appropriate for their products, taking into account that some limitations of use related to Intellectual Property may apply to certain aversive agents, films or technologies.

A7.1.10 It is advised to foresee a safety margin so that the effectiveness of the aversive agent is maintained during the whole life cycle of the product.

A7.1.11 Companies will need to document the levels of aversive agent used and the rationale, and **keep such records for 10 years** (in line with the general REACH and CLP record keeping deadlines).

A7.1.12 Further, the Soluble Packaging Regulation requires the **effective concentration of aversive agent to be safe**. A.I.S.E. recommends to determine that the concentration chosen is safe in case of ingestion of the amount of film contained in one capsule, by means of a human health toxicological risk assessment, based on the highest level of aversive agent contained in the soluble packaging at any time of the product life cycle and adapted to the target age group (young children, including babies). The safety date sheet of the aversive agent is a useful source of toxicological data but may not be sufficient to run a full risk assessment.

[5] Julie A. Mennella, M. Yanina Pepino, and Danielle R. Reed. Genetic and environmental determinants of bitter perception and sweet preferences. *Pediatrics*, 2005, 115 (2), e216–e222.

A7.1.13 Environmental safety should also be documented. It should be reminded that the REACH Registration is the main mechanism to assess environmental safety of substances and demonstrate the use is safe (unless a particular substance does not need to be registered by law). Annex A10 provides an example of a screening environmental risk assessment for one particular aversive agent (denatonium benzoate) showing that, even under conservative assumptions, the addition of this bittering agent in unit dose soluble films is of no concern from an environmental perspective.

A7.2 Capsule Integrity

A7.2.1 Two specific requirements apply under the Soluble Packaging Regulation in relation to capsule integrity: mechanical resistance and liquid containment.

A7.2.2 Both the mechanical and the containment function tests are understood as **'design tests'**. They serve a safety purpose in the qualification of products/validation of processes. They are not considered as quality control tests since it is impossible in practice to test every single capsule.

A7.2.3 These tests should be performed on an appropriate, representative number of capsules at design stage and should be repeated, at the minimum, at every substantial design change in product, film specification, formulation or manufacturing process.

A7.2.4 The capsules will be tested at least 24 h after production after having been conditioned in an environment with a standard temperature and relative humditiy. More details are provided in the test protocols (Annex A11 and Annex A12).

A7.2.5 *Liquid Containment Function:*

A7.2.5.1 The Soluble Packaging Regulation requires the soluble packaging to retain its liquid content for at least 30 s when the capsule is in contact with water. Some of the testing parameters are set by the Regulation (water, temperature).

A7.2.5.2 To A.I.S.E.'s knowledge, no standard method exists for such type of test.

A7.2.5.3 Building on the experience from its members, A.I.S.E. has developed a **containment function test protocol**, which is provided in Annex A11 to this document.

A7.2.6 *Mechanical Integrity:*

A7.2.6.1 The Soluble Packaging Regulation requires the soluble packaging to resist a mechanical compression strength of 300 N under standard test conditions.

A7.2.6.2 A.I.S.E. recommends running a **dynamometric test**: the purpose of such compression test is to assess the mechanical integrity of a capsule submitted to a compressive strength.

A7.2.6.3 Building on the experience from its members, A.I.S.E. has developed a test protocol, which is provided in Annex A12 to this document.

A8. STUDY PROTOCOL: ASSESSMENT OF THE EFFECTIVENESS OF AN AVERSIVE AGENT IN SOLUBLE FILM FOR LIQUID LAUNDRY DETERGENT CAPSULES

A8.1 Objective

A8.1.1 The objective of this test is to determine the effectiveness of a given aversive agent contained in a given soluble packaging film. The dose-response relationship of the level of aversive agent with the observed oral rejection time is investigated. From this, the level of aversive agent that is expected to lead to a rejection time below 6 s is determined.

A8.2 General Study Description

A8.2.1 The response of test panelists to tasting water-soluble film with different levels of aversive agent is to be observed. From this, a dose-response relationship is to be established that links the deterring effect (rejection of the film) with the level of the aversive agent.

A8.2.2 The test panel shall consist of young adults, as a proxy for the target audience for the safety measures on liquid laundry detergent capsules (that is, young children). There are reliable indications that, especially for bitter taste, children are usually more sensitive than adults.

A8.2.3 The test product is the water-soluble film containing (different levels of) the aversive agent. The film shall be used in isolation for tasting: actual detergent capsules shall not be used, to ensure the safety of the panelists.

A8.2.4 Each panelist, unaware of what to expect, will be given a sheet of the soluble film containing a given level of aversive agent, and will be asked to lick the film to experience the taste. It will then be recorded whether the panelist rejects the film and if so, after how much time. panelists are only allowed to participate once, to avoid any bias due to prior experience with a bad tasting sample.

A8.2.5 A concentration series will be tested, in two rounds. First, in a screening round, a broad range of levels of aversive agent in film shall be assessed, as well as an untreated blank. Subsequently, based on the screening round results, suitable aversive agent test levels shall be defined for a definitive testing round, aiming to refine the dose-response relationship for those levels leading to a rejection time close to the target of maximum 6 s.

A8.3 Test Material

A8.3.1 The test material is a combination of one specific water-soluble film type with one specific aversive agent, at different concentration levels. Both the water-soluble film and the aversive agent tested shall be identified in the study report and/or in the study sponsor's confidential study placement documentation. The results of the study are specific to the type/grade of water-soluble film and the type/grade of aversive

agent used. Consequently, results cannot be extrapolated to substantially different combinations of film and aversive agent[6].

A8.3.2 *Preparation of Water-Soluble Film Treated with Aversive Agent:*

A8.3.2.1 Water-soluble films with different levels of the aversive agent shall be prepared:

(1) Screening test: untreated (blank) – 10ppm – 100ppm – 1000ppm – 10000ppm (*)(**)

(*) a toxicological safety assessment shall be conducted prior to the study. if toxicological concerns exist with the highest screening levels, an alternative concentration series with lower levels should be used.

(**) a range with a different upper level may be used if pre-existing information suggests this is more appropriate.

(2) Final test: 6 levels (no blank) to be determined based on the outcome of the screening test.

A8.3.2.2 Accuracy of the aversive agent's levels in the film, and homogeneity of its distribution, shall be ensured by the producer of the treated film.

A8.3.3 *Preparation of the Film Sheets For Taste Testing:*

A8.3.3.1 The treated water-soluble films shall be cut into strips of 3cm by 10cm. For each test concentration, at least 12 replicates shall be prepared. The strips of film shall then be placed in individual bags, to ensure contamination is not an additional variable for the study.

A8.3.3.2 Subsequently, for each concentration, the strips shall be split into two equal batches—one batch for male panelists, one batch for female panelists. The sets of test specimens for female and male panelists shall be kept separate and identified as such.

A8.3.3.3 These test specimens shall be individually labelled using a coding system that links the specimen to its aversive agent level. The coding shall not disclose the level of aversive agent neither to the panelists, nor to the persons directly handing the test specimens to the panelists. This is to avoid any bias, by applying a double-blind approach. For the same reason, preparation, packing and labelling of the film strips shall be done by different persons than those conducting the study with the panelists.

A8.4 Test Panel

A8.4.1 A test panel with as many participants as there are test specimens (that is, in total 10 test concentrations + one blank, with minimum 12 replicates each, hence a total of at least 132 panelists) is required to conduct this study for one film/aversive agent combination.

A8.4.2 For ethical considerations, the test panel shall not consist of young children, but instead, as a proxy, young adults shall be used. It should be noted that this is expected to lead to some difference in the results, as adults tend to 'think' about the bad taste that is happening rather than react and spit it out. The study has been designed to eliminate as much of the adults 'over thinking' to the test as possible, attempting to gage a 'true' reaction time.

A8.4.3 The test panel shall consist of the following individuals:

A8.4.3.1 young adults, in the age group of 18-25 years old

A8.4.3.2 equal mix male/female

A8.4.3.3 exclusion criteria:

(1) smokers shall be excluded.

(2) panelists with prior experience on tests of aversive agents in this context shall be excluded.

A8.4.4 Each panelist shall participate to only one single tasting session, to avoid a biased response driven by prior experience.

A8.5 Test Design and Instructions

A8.5.1 The test shall be conducted in two rounds:

A8.5.1.1 a screening round in which a wide range of levels of the aversive agent is assessed;

A8.5.1.2 a final round in which the dose-response relation close to the rejection time target is refined.

A8.5.2 In the screening round, there shall be 4 test concentrations in addition to a blank (untreated film). In the final round, there shall be 6 test concentrations, and no blank. There shall be at least 12 replicates for each concentration. Hence, in total, there will be at least 132 tasting sessions (5×12=60 for the screening round, and 6×12=72 for the final round). If deemed necessary based on the results of the screening round, a higher number of replicates may be used for the final round.

A8.5.3 The levels of aversive agent for the screening round are predetermined. The levels for the final round are to be defined based on the screening results. Consequently, the final round can only be organized several weeks after the screening round, to allow for processing of the screening data, and for preparation and shipment of the film and test specimens.

A8.5.4 For the actual testing, the test specimens shall be provided to the person conducting the study in two batches: one for female panelists, and one for male panelists. As outline above, each of these batches shall contain an equal number of replicates for each test concentration. Consequently, every test concentration shall be tested with an equal number of males and females, to avoid any potential bias driven by the panelists' gender.

A8.5.5 For every tasting session (one panelist, one level of the aversive agent) the following method shall be followed:

A8.5.5.1 The test shall be conducted such that participating panelists cannot see the reaction of others in the test, and cannot talk to others who have just completed the test. The panelists shall not be informed about the presence of an aversive agent in the sample. The persons providing the test samples to the panelists shall not be informed about the level of the aversive agent in the sample.

A8.5.5.2 The panelist shall drink a defined small amount (50 ml) of still water.

A8.5.5.3 The following exact instructions shall be given to the panelist: "This is a taste test and it is what we call

[6] It is also up to each company to select the aversive agent they deem appropriate for their products, taking into account that some limitations of use related to Intellectual Property may apply to certain aversive agents, films or technologies.

'double-blind', meaning I do not know what taste you are going to receive. It could be anything from a neutral non-taste to something pleasant or unpleasant, it could be *salty, sweet, acidic*[7] etc. If, when you are licking it, you think the taste is neutral or pleasant, I want you to continue licking it until I tell you to stop. If, when you are licking it, you discern that the taste is something unpleasant, I want you to stop licking it immediately. You are going to take the film that is in the bag and hold it in your hands and lick it like so..." and then the panelist will be shown how to hold and lick the film.

A8.5.5.4 At the moment of contact of the film strip with the tongue/mouth, a timer shall be started, and no further instructions shall be provided to the panelist. Each panelist's reaction may be filmed for future reference.

A8.5.5.5 It shall be recorded whether the panelist rejected the test specimen prior to the strip's dissolution in half, and if so, exactly after how many seconds the rejection occurred.

A8.5.5.6 Participants shall be given something to eat or drink to remove the bad taste. What is to be offered will depend on the aversive agent under study. For example, for bittering agents, strong dark chocolate is known to effectively remove the bitter taste. In addition, flavoured lip balm shall be offered in case the bad flavour has travelled to the lips of the panelists.

A8.5.5.7 Exclude the panelist from any further participation to this test or similar tests in the future.

A8.6 Analysis and Reporting of Results

A8.6.1 All raw data collected during the study shall be reported, except for the identities of the panelists (that are to remain confidential to the testing laboratory). Note that these identities shall be archived by the testing laboratory for further reference, to avoid their participation in other similar studies in the future.

A8.6.2 Among the panelists, it is expected that there will be a natural variability in taste receptor sensitivity, primarily driven by genetic differences. People who lack sensitivity in the receptors that are targeted by a specific aversive agent, will experience the aversive taste to a limited extent, if at all (irrespective of the concentration of the aversive agent). For example, in Sibert & Frude (1991)[8] in a test where children were given orange juice spiked with a common aversive agent (denatonium benzoate) at a level known to be effective, over 15 % of the test subjects showed no evident response.

A8.6.3 The aim of the study is to determine the appropriate aversive level that leads to oral rejection within 6 s of the initial exposure. For non-sensitive subjects, this rejection time cannot be achieved, irrespective of the level of aversive agent used. Consequently, data from non-sensitive subjects should be ignored when determining the appropriate level. Hence, the median of the different replicates at a given level shall be used as the relevant metric for comparison with the 6-s target.

A8.6.4 By means of suitable statistical methods (to be determined case-by-case, depending, for example, on the distribution shape and amount of scatter of the data) it shall be determined which levels of aversive agent have led to a median rejection time below the target of 6 s, with at least 90 % confidence. If feasible (depending on the quality of the data), a mathematical dose-response relationship shall also be developed, that allows to determine rejection time as a function of the aversive agent level. Furthermore, it shall be determined up to which level of aversive agent the observed rejection time is not significantly different from the blank; and as of which level of aversive agent the observed rejection time no longer decreased.

A8.6.5 The final outcome of the study is the determination of the lowest aversive agent level that is expected to lead to a median rejection time (either observed as tested; or calculated if a mathematical dose-response relationship could be developed) below 6 s, with at least 90 % confidence.

[7] The actual description of the taste of the aversive agent under study shall not be used here. For example if a bittering agent is used, the word 'bitter' shall not be mentioned; if the aversive agent has an acidic taste, the word 'acidic' shall not be used, etc.

[8] Sibert J. R. Frude N. (1991). Bittering agents in the prevention of accidental poisoning: children's reactions to Denatonium Benzoate (Bitrex). *Archives of Emergency Medicine*, 1991, 8, 1-7.

A9. SUMMARY OF INTERTEK STUDY FINDINGS: "ASSESSMENT OF THE EFFECTIVENESS OF AN AVERSIVE AGENT IN SOLUBLE FILM FOR LIQUID LAUNDRY DETERGENT CAPSULES"

A9.1 Executive Summary

A9.1.1 The proposed test method to assess the effectiveness of an aversive agent in soluble film for liquid laundry detergent capsules was found to be practically feasible, and to allow defining an aversive agent's effective level in the context of Commission Regulation (EU) No 1297/2014.

A9.1.2 It is recommended to use the median oral rejection time as the appropriate metric to assess compliance with the requirements. Non-parametric statistical methods are needed, because the rejection times between panelists are not normally distributed. Specifically, a certain percentage of the population is typically less or not sensitive to a given aversive agent due to natural variability (genetic predisposition), which leads to skewed distributions and scattered observational data. This implies that a sufficient number of replicates (at least 12 but ideally more) is required per tested level of aversive agent, to ensure robustness of the results.

A9.1.3 For one specific grade of PVA film, treated with the bittering agent denatonium benzoate, a dose-response relationship was observed with a decreasing rejection time up to 220 ppm. The rejection time remained the same when the aversive agent's level was further increased beyond this level. The

median rejection time for levels>= 220 ppm was 2.7 s, and was demonstrated to be significantly less than 6 s with >95 % confidence.

A9.2 Background

A9.2.1 Commissioned by A.I.S.E., Intertek carried out a study to measure the reaction time of young adults when coming into oral contact with soluble film treated with an aversive agent. The response of the test panelists to tasting water-soluble film with different levels of aversive agent was observed. From this, a dose-response relationship was established that links the deterring effect (rejection of the film) with the level of the aversive agent.

A9.2.2 The objectives of this study were twofold:

A9.2.2.1 the development of a method for measuring the oral rejection time by young adults, as a function of the level of aversive agent present in water-soluble film of detergent capsules; and

A9.2.2.2 proof of the concept with one specific commonly-used aversive agent and one specific film.

A9.2.3 For the method development, a pilot study was conducted with internal Intertek employees. Next, through a screening study with 50 panelists (5 tested levels, 10 replicates each), it was determined what are the appropriate levels of the aversive agent to be tested in more detail. A final study was then conducted with 72 panelists (6 tested levels, 12 replicates each). A follow-up study with orange juice that was spiked with the aversive agent, was conducted afterwards with 10 panelists, to assess whether these may have been non-sensitive to the aversive agent. All these studies were conducted at the Intertek facility in Oak Brook, Illinois in the US.

A9.3 Method Development

A9.3.1 Overall, it can be concluded that the developed test protocol is practically feasible and that it can be used to determine the effective level of an aversive agent leading to rejection within a defined time period.

A9.3.2 The most suitable method of delivery was found to be a sheet of film (3×10cm), to be licked by the panelists until discerning that the taste is something unpleasant. Clear wording was developed to have unambiguous instructions for the panelists. This was well understood (with only 1 exception out of 132 panelists).

A9.3.3 To rule out any difference due to different taste sensitivities between males and females, both genders should be equally represented and each gender group should receive the same distribution of aversive agent levels tested.

A9.3.4 The observed rejection times (especially for those aversive agent levels that lead to a substantial repulsive effect), were found to not follow a normal distribution. A majority (75 to 80 %) was clustered around a short rejection time, while the remainder was very scattered. This is directly driven by the biology: genetically, a certain part of the population has less (or no) effective receptors for the specific aversive taste. As such, it can be anticipated that similar distribution shapes may be found with other aversive agents and/or other soluble films than the ones used for the method development. It should be noted that follow-up to assess possible non-sensitivity of panelists with long rejection times was not found to add substantial value. Instead, appropriate statistical methods should be used that implicitly take into account the 'biological outliers'.

A9.3.5 As a consequence of the non-normality, the use of mean rejection time is not relevant. Instead, the median should be used, as this is independent of the distribution shape at its extremes. Using Sign Analysis (a non-parametric method) it can be assessed whether the observed median is significantly below the required threshold of 6 s, with a given level of statistical confidence (for example, 90 or 95 %).

A9.3.6 Another consequence of the non-normality is that a sufficiently high number of replicates is required for each tested level. 12 replicates per level, as applied in the final round of this study, is judged to be a minimum. But a larger number of replicates is to be preferred, to increase statistical robustness.

A9.4 Proof of concept for a specific PVA film containing Denatonium Benzoate

A9.4.1 As a proof of concept, the method was applied to determine the required effective level of one specific aversive agent (a bittering agent: denatonium benzoate) selected based on its commonality and one specific polyvinyl alcohol (PVA) film grade (Monosol M8630).

A9.4.2 No reduction of the oral rejection time versus untreated film was seen up to 10 ppm of denatonium benzoate in the film. At 50 ppm, a clearly lower rejection time was observed, and this further decreased at 110 ppm and again at 220 ppm, where a median value of less than 3 s was reached. Higher levels did not cause the median rejection time to drop further. The dose-response relationship is shown in the below chart. Please note that for the ppm levels a logarithmic scale was used. The data shown are from the final study except the data points in red (screening study). (See Fig. A9.1.)

NOTE A9.1—The median of 9.8 s observed in the screening round for 1000 ppm is judged to be on artifact caused by the too limited number of replicates. When the observed rejection times for 1000 ppm (screening round) and those for the very similar level of 960 ppm (final round) are grouped, the median is 3 s.

A9.4.3 The observed dose-response relationship is statistically supported by the Mann-Whitney test. This shows that the rejection times at the higher levels were not significantly different from those at 220 ppm (that is, flat dose-response beyond 220 ppm). Further, this test shows that all treatment levels in the final study led to significantly lower rejection times than the 0 ppm blank, and that the rejection time at 220 ppm was significantly less than at 110 ppm. Finally the test shows that the rejection time at 10 ppm (screening round) was not less than for the blank.

A9.4.4 The median oral rejection time for denatonium benzoate levels in film ≥220 ppm (in the final study) was on average 2.7 s. Sign analysis shows that for each of these levels, the median was significantly below 6 s, with a confidence level of >95 %. The 75th percentile of the observed rejection times was also below 6 s for all levels ≥220 ppm (in the final study), however, statistical significance could not be demonstrated.

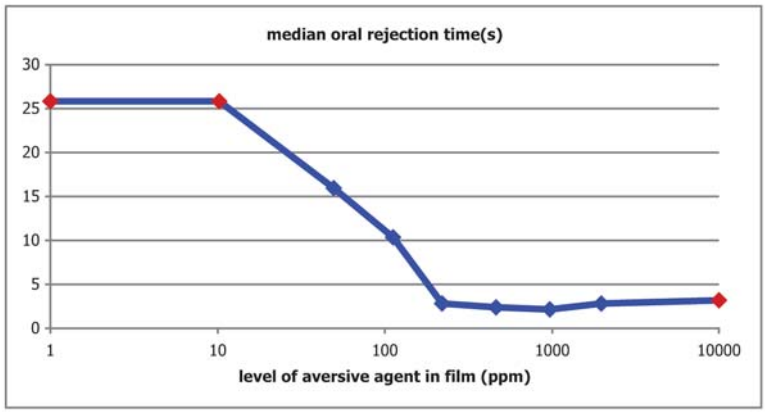


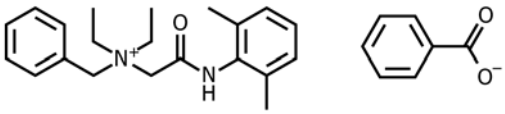
FIG. A9.1 Final Study Data

A9.4.5 It can be concluded that, for the specific film grade that was tested, a denatonium benzoate level of 220 ppm in the film is adequate to meet the requirements of Commission Regulation (EU) No 1297/2014.

A10. SCREENING ENVIRONMENTAL RISK ASSESSMENT FOR DENATONIUM BENZOATE (EXAMPLE OF AVERSIVE AGENT)

A10.1 Substance Identification

A10.1.1 Denatonium benzoate is a salt of the quaternary ammonium cation denatonium with the inert anion benzoate:



CAS	3734-33-6
Molecular formula	$C_{28}H_{34}N_2O_3$
Molar mass	446.581

A10.2 Environmental Properties

A10.2.1 *Ecotoxicity:*

A10.2.1.1 In the European Classification & Labelling notification process (ECHA, 2015), denatonium benzoate was notified as Aquatic Chronic 3 (H412) by most notifiers.

A10.2.1.2 In the EU Ecolabel DID LIST (European Commission, 2014), denatonium benzoate is included (ingredient nr. 2604). As the relevant acute LC50, a value of 13 mg/L is mentioned. Chronic data are absent.

A10.2.1.3 The following ecotoxicologal data are reported in several safety data sheets (from multiple suppliers) of denatonium benzoate, and/or in regulatory reviews (for example, US CPSC 1992; Health Canada, 2011):

(1) Fish: 96h LC50 Rainbow Trout: >1000 mg/L

(2) Invertebrates: 96h LC50 Shrimp (salt water): 400 mg/L

(3) Invertebrates: 48hr EC50 Daphnia magna: 13 mg/L

(4) No effects on bacteria up to 150 mg/L

A10.2.1.4 The Predicted No-Effect Concentration (PNEC) can be derived from the lowest acute data point, in this case for the water flea Daphnia magna (which is also the value used for the EU ecolabel). The assessment factor to extrapolate from an acute EC50 to the ecosystem safe level is a factor 1000. Hence, the PNEC = 13 µg/L.

A10.2.2 *Biodegradability:*

A10.2.2.1 The active cation denatonium was not found to be either biodegraded or adsorbed to sludge in a Semi-Continuous Activated Sludge (SCAS) study (Corby et al., 1993). As a SCAS test simulates fate in actual sewage treatment plants, it is fair to assume that denatonium benzoate will not be removed in sewage treatment.

A10.2.2.2 Furthermore (cf. CPSC, 1992), in an OECD 301D test, no chemical deterioration of Denatonium benzoate was observed. In the Zahn-Wellens test (OECD 302B), a 36 % breakdown was found after 28 days. A carbon dioxide production test showed that denatoinum benzoate is poorly metabolized (4.5 % after 28 days).

A10.2.2.3 In the EU Ecolabel DID LIST, denatonium benzoate is assumed to be not removed in sewage treatment (DF=1).

A10.2.3 *Bioaccumulation*—Denatonium benzoate is highly water soluble (45 g/L) and has a low octanol/water partitioning coefficient (Kow = 0. 91) (cf. Health Canada, 2011). Consequently, there is no risk for bioaccumulation.

A10.3 Environmental Risk Assessment

A10.3.1 *Tonnage Estimate:*

A10.3.1.1 As it has not yet been registered under REACH, the tonnage of denatonium benzoate across the EU is in the order of <100 ton per year per legal entity. For the purpose of this screening assessment one could conservatively assume a total of 100 ton per year, which is equivalent to 200 mg per capita per year in the EU (with 500 million people).

A10.3.1.2 The incremental consumption of denatonium benzoate in the context of liquid laundry detergent capsules, can be estimated as follows:

(1) One laundry capsule of 5cm × 5cm with a film thickness of 100 µm has 5 × 5 × 0.01 × 2 sides = 0.5 cm^3 of soluble film as outer packaging. With a density of 1.3 this corresponds to 0.65 g of film per capsule.

(2) When 200 [respectively 1000] ppm is used as aversive agent contained in the film, this leads to the presence of 130 [resp. 650] µg of denatonium benzoate per capsule.

(3) In the United Kingdom, which is to date the most mature market for laundry capsules, on average about 20 capsules are sold per year per inhabitant (total market: 1150 million capsules; population of 64 million).

(4) This corresponds to 2.6 [resp. 13] mg of denatonium benzoate per capita per year.

A10.3.1.3 Consequently, an assessment of the assumed current tonnage of 100 ton/year in the EU (= 200 mg/cap.year) covers any potential increase due to the introduction of denatonium benzoate in the soluble film of laundry capsules at the envisaged levels.

A10.3.2 *Risk Assessment:*

A10.3.2.1 The average water use per person per year in the EU is 100-200 L per capita per day (EEA web site). Conservatively, a water use of 100 L/day is assumed.

A10.3.2.2 Assuming 100 ton/year in the EU, the concentration of denatonium benzoate in household waste water is 200 mg/cap. year divided by 365 days/year × 100 L/cap.day = 5.5 µg/L. As denatonium benzoate is not removed in sewage treatment plants, this is also the predicted concentration for treated effluent. Finally, the Predicted Environmental Concentration (PEC) in river water, taking into account a standard dilution factor of 10, is 0.55 µg/L.

A10.3.2.3 The aquatic PNEC for denatonium benzoate is 13 µg/L (derived from Daphnia magna acute data with an assessment factor of 1000).

A10.3.2.4 The PEC/PNEC ratio for denatonium benzoate is 0.55 / 13 = 0.04. In other words, the calculated safety margin is by a factor >20. It should be noted that this is based on a conservative tonnage estimate of total consumption, which is also nearly two orders of magnitude higher than the expected use of this substance for laundry capsules.

A10.4 Conclusion

A10.4.1 Using conservative assumptions, especially regarding tonnage, this screening assessment shows no concerns with the environmental safety of denatonium benzoate. The incremental use of denatonium benzoate as aversive agent in laundry detergent capsules is minimal compared to the assumed total tonnage, and is not anticipated to negatively impact this conclusion.

A10.5 References

Corby, J., Doi, J., Conville, J., Murphy, S. et al., "Biodegradability of a Denatonium Bitterant," SAE Technical Paper 930587, 1993, doi:10.4271/930587.

European Chemicals Agency http://echa.europa.eu. Accessed 7.1.2015.

European Commission (2014). Detergents Ingredients Database, version 2014.1 http://ec.europa.eu/environment/ecolabel/documents/did list/didlist part a en. pdf

European Environment Agency http://www.eea.europa.eu/themes/households Accessed 7.1.2015.

Health Canada (2011). Proposed Re-evaluation Decision PRVD2011-15 Denatonium Benzoate 08 November 2011. http://www.hc-sc.gc.ca/cps-spc/altformats/pdf/pubs/pest/decisions/rvd2012-06/rvd2012-06-eng.pdf

US Consumer Product Safety Commission 1992. Study of Aversive Agents https://www.cpsc.gov//PageFiles/96066/aversive.pdf

A11. CONTAINMENT FUNCTION TEST PROTOCOL

A11.1 This test is a design test. It serves a safety purpose for the qualification of products and the production process.

A11.2 This test shall be performed on an appropriate, representative number of capsules and repeated, at the minimum, at every substantial design change in product, film specification, formulation or manufacturing process.

A11.3 Sample Conditioning Prior to Testing

A11.3.1 Capsules shall be tested after having been conditioned at **23 ± 1°C/50 ± 2 % Relative Humidity** for at least **24 h** in the original outer packaging opened to the conditioning atmosphere.

A11.3.2 These conditions are in line with Practice D4332.

A11.4 Test Method

A11.4.1 A beaker of sufficient capacity is filled with at least 1 L of demineralised water.

A11.4.2 Once the temperature has stabilised at 20°C, one pre-conditioned capsule is gently introduced into the beaker until it is entirely submerged by water. The capsule shall be surrounded by water on all sides.

A11.4.3 In case the density is such that the capsule either floats or sinks, the capsule shall be placed inside a device that prevents floating or sinking, such as a metal cage, a netting bag or a similar device that allows visual observation.

FIG. A11.1 Stages of Containment Loss

A11.5 Recording Containment Loss

A11.5.1 A timer shall be started as soon as the capsule is submerged by water.

A11.5.2 The dissolution of the capsule shall be observed visually as a function of time, with the following event recorded: "Liquid Content Release," which corresponds to the first visual evidence of liquid leaving the capsule. To successfully pass the criteria, the time recorded once liquid content released is observed should be at least 30 s.

A11.5.3 The following pictures (see Fig. A11.1) visually illustrate the observable stages of containment loss (these experiments were not strictly conducted according to the above protocol and visual observations may differ depending on the capsule design, colour size or shape. These pictures are only for illustrative purpose).

A11.5.3.1 Prior to product release with fully closed containment.

A11.5.3.2 The moment in time when **first release of product occurs is noted**.

A11.5.3.3 Further progress of product release and air escapes from capsule.

A11.6 Criteria for Passing the Test

A11.6.1 In line with general principles of testing of safety-related features (such as ISO 8317), the test will be successful when at least 85 % of the capsules tested do not release their content within minimum 30 s, with a 90 % confidence level.

A11.7 Experimental Design

A11.7.1 A.I.S.E. recommends applying one of the two following methods to **determine whether the content release time is at least 30 s, for 85 % of the capsule, with 90 % confidence**.

A11.7.1.1 *Attribute Test, Non-destructive*—The test is conducted for exactly 30 s for each capsule that is tested. Capsules not releasing their content within this period of time are recorded as successful, capsules that release content before 30 s are counted as failures. By means of binomial statistics,[9] it can be determined how many failures are allowed as a function of the total number of samples tested, to achieve an overall 85 % success rate with 90 % confidence:

Number of samples tested	Number of failures allowed (85 % success rate at 90 % confidence)
< 15	No valid test possible
15-24	0
25-33	1
34-42	2
43-51	3
52-59	4
60-67	5
68-76	6
77-84	7
85-92	8
93-100	9

NOTE A11.1—The above table can only be used in the context of design testing, to ensure with 90 % statistical confidence that 85 % of the samples will meet the criteria. The table is not applicable for other purposes such as inspections, for which different statistical criteria need to be applied.

A11.7.1.2 *Determining the Content Release Time by Destructive Testing*—The test is conducted until the liquid content starts to be released, and this time is recorded for each capsule in the test. From these data, a statistical distribution is constructed. Using appropriate statistical methods, that depend on the shape of the observed distribution, it can be determined whether 85 % of the population of samples will have a content release time greater than 30 s, with 90 % confidence. The information can also be used to optimise the number of samples required for future testing.

[9] Gilliam, D., Leigh, S., Rukhin, A., and Strawderman, W., "Pass-Fail Testing: Statistical Requirements and Interpretations," *J. Res. Natl. Inst. Stand. Technol*. 114, 195-199 (2009).

A12. DYNAMOMETRIC TEST PROTOCOL

A12.1 This test is a design test. It serves a safety purpose for the qualification of products and the production process.

A12.2 This test shall be performed on an appropriate, representative number of capsules and repeated, at the minimum, at every substantial design change in product, film specification, formulation or manufacturing process.

A12.3 Sample Conditioning Prior to Testing

A12.3.1 Capsules will be tested after having been conditioned at **23 ± 1°C/50 ± 2 % Relative Humidity** for at least **24 h** in the original outer packaging opened to the conditioning atmosphere.

A12.3.2 These conditions are in line with Practice D4332.

A12.4 Test Method

A12.4.1 One capsule is submitted to an increasing compression force at a rate of 200 to 250 mm/min (typically in the range of operation of standard equipment) until 300 N is reached or until it releases its content, under standard test conditions.

A12.4.2 'Standard test conditions' in this context refers to test conditions which are similar to the conditioning atmosphere of capsules (see previous paragraph). Therefore, capsules shall be tested shortly after having been sampled from the conditioning atmosphere.

A12.4.3 The instrument is made of two flat plates of a surface larger than the surface area of the capsule.

A12.4.4 The capsule is to be placed in a plastic bag to avoid spillage and positioned between the two plates that apply the force, resting on its largest surface area.

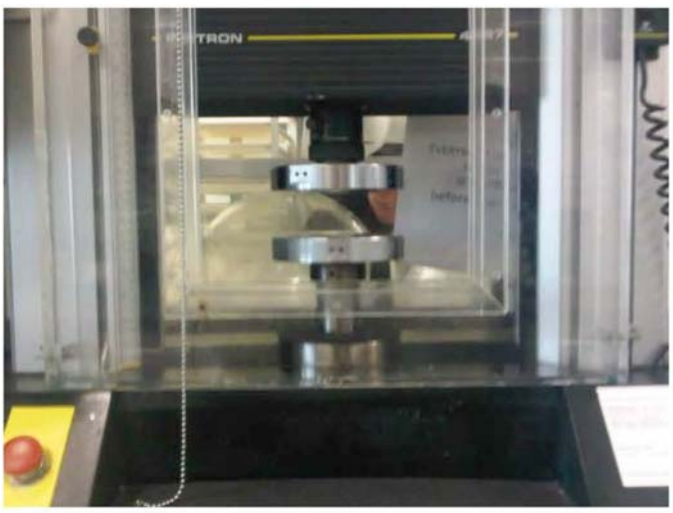


FIG. A12.1 Testing Instrument

A12.4.5 The type of instrument used for such testing is shown in Fig. A12.1.[10]

A12.5 Criteria for Passing the Test

A12.5.1 In line with general principles of testing of safety-related features (such as ISO 8317), the test will be successful when at least 85 % of the capsules tested resist a mechanical compression of 300N, with a 90 % confidence level.

A12.6 Experimental Design

A12.6.1 A.I.S.E. recommends applying one the two methods to determine whether 85 % of the capsules resist against a mechanical compression of 300 N, with 90 % confidence.

A12.6.1.1 *Attribute Test, Non-destructive*—The test is conducted until a compression strength of exactly 300 N is reached for each capsule that is tested. Capsules resisting this compression are recorded as successful, capsules that burst before 300 N is reached are counted as failures. By means of binomial statistics,[9] it can be determined how many failures are allowed as a function of the total number of samples tested, to achieve an overall 85 % success rate with 90 % confidence:

Number of samples tested	Number of failures allowed (85 % success rate at 90 % confidence)
< 15	No valid test possible
15-24	0
25-33	1
34-42	2
43-51	3
52-59	4
60-67	5
68-76	6
77-84	7
85-92	8
93-100	9

NOTE A12.1—The above table can only be used in the context of design testing, to ensure with 90 % statistical confidence that 85 % of the samples will meet the criteria. The table is not applicable for other purposes such as inspections, for which different statistical criteria need to be applied.

A12.6.1.2 *Determining the maximal compression strength by destructive testing*—For each capsule, the mechanical compression strength shall be gradually increased at the rate mentioned above until the capsule breaks. The strength value applied at this point of breakage shall be recorded. The distribution curve (number of samples versus strength applied at the break point) shall be constructed. Using appropriate statistical methods that depend on the shape of the observed distribution, it can be determined whether 85 % of the population of samples will have a compression resistance greater than 300 N, with 90 % confidence. The information can also be used to optimise the number of samples required for future testing.

[10] The sole source of supply of the apparatus (Instron model 5566) known to the committee at this time is Instron, 825 University Ave, Norwood, MA, 02062-2643, www.instron.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.