INTERNATIONAL STANDARD

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Packaging — Transport packaging for dangerous goods — Plastics compatibility testing

Emballages — Emballages pour le transport des marchandises dangereuses — Essais de compatibilité des matières plastiques

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 16101 was prepared by the European Committee for Standardization (CEN) in collaboration with Technical Committee ISO/TC 122, *Packaging*, Subcommittee SC 3, *Performance requirements and tests for means of packaging, packages and unit loads*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Throughout the text of this document, read "...this European Standard..." to mean "...this International Standard...".

Contents

Foreword

This document (EN ISO 16101:2004) has been prepared by CEN /TC 261, "Packaging", the secretariat of which is held by AFNOR, in collaboration with ISO/TC 122 "Packaging".

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2005, and conflicting national standards shall be withdrawn at the latest by March 2005.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports the objectives of the framework Directives on Transport of Dangerous Goods.

This European Standard has been submitted for reference into the RID and/or in the technical annexes of the ADR. Therefore in this context the standards listed in the normative references and covering basic requirements of the RID/ADR not addressed within the present standard are normative only when the standards themselves are referred to in the RID and/or in the technical annexes of the ADR.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

Introduction

This standard was developed to provide requirements and test procedures to meet the compatibility provisions for plastics packagings to contain liquids as set out in:

The European Agreement concerning the International Carriage of Dangerous Goods by Road (ADR) (covering most of Europe) [2] and

Regulations concerning the International Carriage of Dangerous Goods by Rail (RID) (covering most of Europe, parts of North Africa and the Middle East) [5].

This procedure is an alternative option to that set out in the UN Recommendations on the Transport of Dangerous Goods [1].

Plastics packaging material can be attacked by the chemical contents of the package. Such effects are caused by different mechanisms such as environmental stress cracking (ESC) chemical degradation and swelling.

The UN Recommendations and the associated modal regulations require that all packagings shall be assessed for compatibility with the substances which they are to contain. The UN text makes special reference to plastics packagings for liquids. The procedure therein contains details of testing for six months at ambient temperature with the liquid to be carried. RID/ADR permits as an alternative the use of standard liquids to which this document refers.

The UN Recommendations are given legal entity not only to ADR and RID but also to:

The International Civil Aviation Organisations Technical Instructions for the SafeTransport of Dangerous Goods by Air (ICAO Tis) (worldwide) [3] and

The International Maritime Dangerous Goods Code (IMDG Code) (worldwide) [4].

These two modal rules do not refer to the standard liquid tests but they may still be acceptable as the UN provisions do not make the six month test a mandatory requirement.

The application of this standard will need to take account of the requirements of these international agreements and the relevant national regulations [6] [7] for domestic transport of dangerous goods.

Although not stipulated in the UN Recommendations or the modal regulations, these tests may be applied, where deemed appropriate, to inner packagings of combination packagings. However, for this purpose, the standard liquid tests may not be applicable to all types of plastics materials, since the tests were originally created for high molecular weight high density polyethylene (PE-HD-HMW).

WARNING — The use of this International Standard may involve hazardous materials and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This standard specifies the requirements and test methods for compatibility testing of polyethylene based plastics packagings and composite packagings with plastic inners containing liquids. The testing involves storage with the packaged substance, or with a standard liquid as defined in annex A. Annex B describes small scale laboratory tests, which may be used to determine the assimilation of those products to be carried with the standard liquids.

NOTE This standard should be used in conjunction with one or more of the International Regulations set out in the Bibliography

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 291, *Plastics - Standard atmospheres for conditioning and testing*

EN ISO 527-2, *Plastics - Determination of tensile properties - Part 2: Test conditions for moulding and extrusion plastics (ISO 527-2:1993)*

EN ISO 1133, *Plastics - Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics (ISO 1133:1997)*

EN ISO 1183-1, *Plastics - Methods for determining the density of non-cellular plastics – Part 1: Immersion method, liquid pyknometer method and titration method (ISO 1183-1:2004)*

EN ISO 1628-3, *Plastics - Determination of the viscosity of polymers in dilute solution using capillary viscometers - Part 3: Polyethylenes and polypropylenes (ISO 1628-3:2001)*

EN ISO 1872-2, *Plastics — Polyethylene (PE) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties (ISO 1872-2:1997)*

EN ISO 2818, *Plastics – Preparation of test specimens by machining (ISO 2818:1994)*

EN ISO 11403-3 *Plastics - Acquisition and presentation of comparable multipoint data - Part 3: Environmental influences on properties (ISO 11403-3:1999)*

EN ISO 11542-2:1998, *Plastics - Ultra-high-molecular-weight polyethylene (PE-UHMW) moulding and extrusion materials - Part 2: Preparation of test specimens and determination of properties (ISO 11542-2:1998)*

EN ISO 16104:2003, *Packaging – Transport packaging for dangerous goods – Test methods (ISO 16104:2002)*

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)*

ISO 16770, *Plastics - Determination of environmental stress crack (ESC) of polyethylene – Full-notch creep test (FNCT)*

3 Terms and definitions

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

3.1
competent authority
any national regulatory body or authority designated or otherwise recognised as such for any purpose in connection with the regulations specified in the Bibliography

3.2
plastics packagings
drums, jerricans and composite packagings with inner plastics receptacle made from certain types of plastics

NOTE Certain types of polyethylene are listed in **A.3.**

3.3
packaged substance (chemical product)
dangerous liquid with which the packaging is to be filled for transport

NOTE Packagings used for solid packaged substances, which can become liquid at temperatures encountered during transport, should also meet the requirements of packagings for liquids.

3.4
standard liquids
defined liquids that are representative in their effect for a specific kind of interaction between a packaged substance and the plastics packaging

NOTE A full description of the standard liquids can be found in **A.2.**

4 Test requirements

4.1 General

Plastics packagings selected in accordance with clause **5** shall be conditioned with the packaged substance or a standard liquid with which it is to be assimilated. Annex C contains a list of substances assigned to standard liquids

For other chemicals not listed in annex C, small scale laboratory tests (see annex B) shall be used to prove assimilation with standard liquids. The standard liquid chosen shall be at least as aggressive as the substance to be transported. Where the packaged substance to be filled cannot be assimilated with one of the standard liquids, the packaged substance itself shall be used and its specification recorded. In the event that the effect is more aggressive than that of the standard liquids, the six month procedure shall be followed, as given in **7.2**, or alternatively, and with the exception of nitric acid > 55%, the accelerated procedure, as given in **7.3**.

NOTE When the standard liquid is water, proof of chemical compatibility is not required.

4.2 Conditioning

Plastics packagings shall be conditioned in accordance with clause **7** of this standard.

4.3 Post-conditioning inspection

At the end of the conditioning period the packagings shall be inspected for leakage. Where no leakage is apparent testing in accordance with clause **7** of EN ISO 16104:2003 shall commence within 21 days of the end of the conditioning period (see **7.4**).

4.4 Drop test

When tested in accordance with **7.1** of EN ISO 16104:2003, the plastics packaging:

a) shall be leakproof, subsequent to any slight discharge that may be apparent at the moment of impact and subsequent to the equalization of internal and external pressures (except for inner packagings of combination packagings, when it is not necessary for the pressure to be equalized);

b) shall not exhibit any damage liable to affect safety during transport, for example if the package cannot be used without leaking.

4.5 Stacking test

When tested in accordance with **7.2** of EN ISO 16104:2003, the packaging shall not:

a) show any sign of leakage ;

b) show any deterioration which could adversely affect transport safety, nor any distortion liable to reduce its strength or stability in stacks of packages.

4.6 Leakproofness test

When tested in accordance with **7.3** of EN ISO 16104:2003, plastics packagings shall be leakproof.

4.7 Hydraulic pressure test

When tested in accordance with **7.4** of EN ISO 16104:2003, plastics packagings shall be leakproof.

4.8 Permeability testing

With the exception of composite packagings having a plastics receptacle with outer steel drum, when tested in accordance with **8** of this standard, plastics packaging shall have a permeability not exceeding 0,008 g/l.h.

4.9 Equivalent testing

The test methods described in this standard shall be considered to be the reference test methods.

NOTE Alternative methods may be used to demonstrate compliance with relevant regulations provided that:

- their equivalency to the reference test method can be demonstrated;
- their use is recorded in the test report;
- prior approval is obtained from the competent authority.

4.10 Test report

All packaging tests in conformity with this standard shall be the subject of a test report prepared in accordance with annex E of EN ISO 16104:2003. It shall be possible to specifically identify the packaging relative to each test report, either by the retention of uniquely numbered specimens or by inclusion of sufficient photographs and/or drawings with unique references.

5 Selection and preparation of test packagings

5.1 Selection of packagings

A minimum of 15 packagings of each design type (or 18 in the case of a requirement to carry out the tests in **8)**, for each chemical to be tested, shall be selected at random from normal production and submitted for testing.

NOTE 1 For box shaped composite packagings a different number of samples, 14 or 17, may be required.

Packagings shall be:

a) at least 48 h old;

b) marked with a test reference number which shall also be entered on the test record and later used on the test report;

c) individually weighed to establish the tare or the filled mass;

NOTE 2 The form of such weighing may be varied to fit in with whether the packagings have been supplied full or empty to the test station. Where the masses of individual empty packagings are recorded, it is necessary to record only a typical filled mass (or vice-versa).

d) examined for damage etc. which might invalidate the tests;

NOTE 3 The tests set out below should be applied to every design type of packaging, by polymer type and grade.

NOTE 4 For selective testing see EN ISO 16104:2003, annex F.

NOTE 5 For testing with a lesser number of packagings see EN ISO 16104:2003, **4.1** NOTE.

5.2 Information to be provided with packagings

Each packaging type shall be accompanied by specification(s) for that design type (in the appropriate format set out in annex G of EN ISO 16104:2003 and by the following additional information as relevant.

The packaging user (with the assistance, where appropriate, of the packaging manufacturer and the test laboratory) shall identify the packaged substance. In the first instance this process shall consist of identifying the plastics material concerned and its possible interactions, such as swelling, environmental stress cracking (ESC), and molecular degradation.

NOTE 1 The specification forms for plastics packagings should identify the material by polymer type and grade.

NOTE 2 Where tests are carried out using the packaged substance the test report may be applicable for other substances having equivalent or lesser chemical effects.

5.3 Filling of packaging prior to testing

5.3.1 Determination of brimful capacity

A packaging intended to contain liquids shall be filled to not less than 98% of the brimful capacity. The brimful (overflow) capacity is determined for example by:

— weighing the empty packaging including closures (mass empty = *m* kg) and

— weighing the packaging full (mass m kgs brimful = W kg).

The packaging shall be filled with water until the water just overflows and then fitting the closure and any surplus mopped up. No steps shall be taken, e.g. by tilting or tapping the packaging, to enable water to penetrate into a hollow handle or other design feature above the closure.

$$b = \frac{W - m}{d}$$

where

b is the brimful capacity in litres

W is the mass of the packaging when brimful with water in kilograms (kg)

m is the mass of the empty packaging in kilograms (kg)

d is the density of water (1,0 kg/l)

NOTE When the brimful capacity has already been determined by testing to EN ISO 16104:2003, this procedure is not necessary.

5.3.2 Filling of packaging

Filling of packagings shall be carried out in accordance with the following:

a) The packaging shall be filled to not less than 98 % of the brimful capacity (see **5.3.1**).

b) When filling test packagings, at least one packaging shall have its capacity and filling level determined as in c). Further specimens of that design type may be filled using a dipstick calibrated on the first specimen or, in the case of small packagings, by mass or volume. When test specimens are filled by mass with a liquid other than water (e.g. anti-freeze solution), the density of that liquid shall be taken into account, in order to obtain the correct volume of fill (≥ 98 %). Determination shall be made of the total empty packaging mass including the closure(s). This facilitates calculation of stacking loads.

c) The calculation of required volume for testing shall be:

$$C = \frac{(W - m)x98}{100d}$$

where

C is the required volume of water in litres (l)

W is the mass of packaging when brimful with water in kilograms (kg)

m is the mass of the empty packaging in kilograms (kg)

d is the relative density of water (1,0 kg/l)

5.4 Closing packagings

Screw type closures shall be tightened to the torque recommended by the applicant where appropriate, which shall be recorded in the test report.

Closure torque shall not differ from one test to another in the test report. If it is necessary to revise a closure torque following a failure in one test, then all tests shall be completed using that torque setting.

All tests for a particular liquid shall be carried out at the same torque.

NOTE 1 The closure torque may vary for different seals.

NOTE 2 It is not necessary to apply the specified torque during the conditioning period if this affects the subsequent performance of the seal during the packaging testing.

Where vented closures are intended for use in the packaging they shall be fitted for testing. Packagings fitted with vented closures shall be inverted after closing and observed for leakage for a period of 5 min. Leakage from the closure vent shall be regarded as a failure. An alternative method of test instead of inversion is shown in Figure 1, clause 7.1.

6 Facilities for testing

6.1 General requirements

Tests shall be carried out at a testing facility capable of meeting the operational provisions of EN ISO/IEC 17025.

NOTE 1 This does not imply a requirement for the test laboratory to have attained third party certification or accreditation, but if appropriate such external approval may be obtained from either a national accreditation body or from the competent authority.

NOTE 2 Testing staff should have a knowledge of the principles of the dangerous goods regulations, as set out in the UN Recommendations.

6.2 Accuracy of measurement equipment

The accuracy of measuring equipment shall be more precise than the accuracy of the measurements in testing, as specified in **6.3**, unless otherwise approved by the competent authority. The measuring equipment shall be calibrated in accordance with the relevant provisions of EN ISO/IEC 17025.

6.3 Accuracy of measurements in testing

Measurement equipment shall be selected such that individual measurement results including errors in reading and calibration shall not exceed the following tolerances:

Mass in kilograms (kg):	± 2 %
Pressure in kilopascals (kPa):	± 3 %
Distance / length in millimetres (mm):	± 2 %
Temperature in degrees Celsius (ºC):	± 1 °C
Humidity in percentage (%):	tolerances are as specified in particular test methods
Time in minutes (min):	± 3 %
Torque in newton·metres (N·m):	± 3 N m or 10 %, whichever is the greater

NOTE For some measurements, the tolerances may be lower in order to have meaningful measurements e.g. when measuring masses or dimensions of empty packagings.

Where only maximum or minimum values are specified in the text, tolerances are one sided e.g. in **7.3** the conditioning temperature may exceed 40°C, but shall not be less.

6.4 Climatic conditions

There shall be adequate climatic facilities to meet the requirements in Table 1 of EN ISO 16104:2003.

6.5 Impact surfaces for drop tests

The drop test area shall be horizontal and flat, massive enough to be immovable and rigid enough to be non-deformable under test conditions and sufficiently large to ensure that the test package falls entirely on the surface.

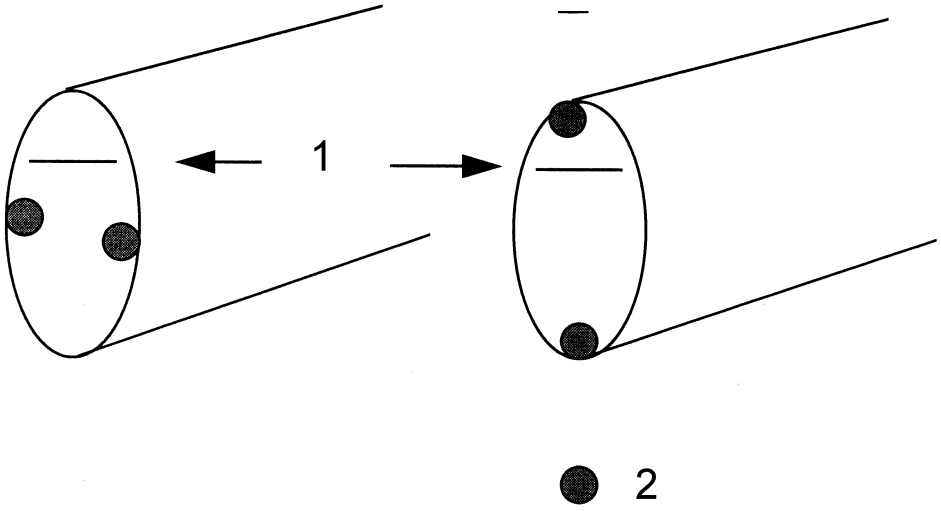
7 Conditioning procedures

7.1 General

After filling, the packagings shall be inverted for 24 h (unless fitted with a vented closure, see following), and then restored to the normal standing position. For the last 24 h of the conditioning period as defined in **7.2** and **7.3**, the packagings shall be inverted again.

For packagings that have vented closures, invert the packagings for a period of 5 min after filling and then restore the packagings to their normal standing position. At the end of the conditioning period, invert the packagings again for a period of 5 min.

NOTE As an alternative to complete inversion the packaging may be laid on its side such that all closures are below the level of the substance being tested (see Figure 1).



Key

1 Liquid level
2 Closure

Figure 1 — Explanatory diagram of alternative inversion method

7.2 Six months ambient conditioning

This test shall be carried out at ambient temperature for a period of 6 months.

For the purposes of this standard, ambient temperature, which shall be monitored and recorded, is considered to be not less than 15 °C.

NOTE The competent authority may, however, allow an extended period of test for temperatures below 15 °C.

7.3 Accelerated conditioning procedure

The packagings for test shall be conditioned for 21 days at a minimum of 40 °C with each standard liquid required.

The procedure shall be applied for polyethylene types as defined in the **A.3.1** and **A.3.2**. For other types of polyethylene such as those defined in **A 3.3** and **A.3.4**, the approval of the competent authority shall be obtained

7.4 Procedure at the end of the conditioning period

At the end of the conditioning period, all packagings, except those intended to withstand the stack test, shall be emptied, rinsed, inspected for damage and prepared for test in accordance with the test procedures for plastics packagings for liquids (EN ISO 16104). Testing shall commence within 21 days of the end of the conditioning period. If emptied the packaging shall be kept closed until testing commences.

Packagings which have been conditioned with standard liquid, n-butyl acetate, shall be emptied and refilled with a mixture of 1 %–10 % aqueous wetting agent solution and 2 % of n-butyl acetate for the stacking test.

NOTE 1 Where the closure elements (for example heat or induction seals) would have to be destroyed to empty the packaging after conditioning; the packaging should be emptied through an additional opening drilled into the package. Such an opening should not affect the results of the other tests (drop, hydraulic pressure and leakproofness tests).

NOTE 2 This does not apply to composite packagings where the outer non-plastics packaging withstands the stacking load, e.g. steel.

NOTE 3 For substances presenting a danger at 40°C it may be necessary to replace the filling substance by another substance where at least the same chemical interaction has been demonstrated and the agreement of the competent authority has been obtained.

The same closures and gaskets used during the conditioning of the packagings shall be used for the rest of the tests; i.e. gaskets and closures shall not be replaced.

7.5 Reuse of standard liquids

The standard liquids shall be checked periodically in accordance with Table 1 as their effectiveness can be reduced over a period of time.

Table 1 — Reuse of standard liquids

Standard Liquid	Specification
Wetting solution	New solution for each test or check surface tension (see annex A)
Acetic acid	Concentration 99 ± 1%
Normal butyl acetate	≥ 98 % [a)]
Mixture of hydrocarbons	16%–21% aromatic content [a)]
Nitric acid	Concentration ≥ 55 %
[a)] It is recommended that the absorption of these standard liquids is periodically checked with a control specimen of polyethylene of defined type and grade, in accordance with **B.4.1** The used standard liquid is no longer fit for purpose when the determined absorption deviates by more than 5 % from the original determined value.	

Tests to monitor the quality of the standard liquids shall be done by appropriate means at intervals according to the frequency of usage.

8 Permeability testing

8.1 Applicability

This test is required only for packagings for benzene, toluene, xylene or mixtures and preparations containing those substances.

8.2 Test preparation

Three packagings shall be preconditioned according to **7.2** of this standard for original filling substance or according to **7.3** of this standard for the standard liquid mixture of hydrocarbons (white spirit).

8.3 Test procedure

The test specimens shall be filled with the packaged substance or standard liquid mixture of hydrocarbons (white spirit) and weighed before and after storage for 28 days at 23°C and 50% relative atmospheric humidity.

NOTE Equivalent procedure at 40°C - this test may be done in conjunction with the accelerated conditioning procedure **7.3** with standard liquid mixture of hydrocarbons (white spirit).

Annex A
(normative)

Standard liquids and applicability to polyethylene types

A.1 Introduction

The standard liquid system has been developed for the investigation of the compatibility of high molecular weight high density polyethylene, but it can also be applied to medium molecular weight polyethylene and to packagings produced from the above polyethylene types where the surface or surfaces have been fluorinated.

NOTE When closures or closure elements are manufactured from materials other than those referred to in **A.3**, alternative suitable methods to investigate compatibility may be employed.

A.2 Definitions and applicability of standard liquids

A.2.1 Wetting solution

Wetting solution shall be used for substances causing severe cracking in polyethylene under stress, in particular for all solutions and preparations containing wetting agents.

An aqueous solution of (1–10)% of a wetting agent shall be used. The surface tension of this solution shall be (31–35) mN/m at 23 °C.

The stacking test shall be carried out on the basis of a relative density of not less than 1,2.

If adequate chemical compatibility is proved with a wetting solution, a compatibility test with acetic acid is not required.

NOTE In the case of filling substances, however, which cause polyethylene to stress crack more than wetting solution, adequate chemical compatibility may be proved after preliminary storage for 21 days at 40 °C in accordance with **7.3** of this standard, using the original filling matter.

A.2.2 Acetic acid

Acetic acid shall be used for substances and preparations causing cracking in polyethylene under stress, in particular for monocarboxylic acids and monovalent alcohol, acetic acid of (98–100) % concentration shall be used with a relative density = 1,05.

The stacking test shall be carried out on the basis of a relative density not less than 1,1.

NOTE In the case of filling substances causing polyethylene to swell more than acetic acid and to such an extent that the polyethylene mass is increased by up to 4 %, adequate chemical compatibility may be proved after preliminary storage for 21 days at 40 °C, in accordance with **7.3**, using the original filling matter.

A.2.3 Normal butyl acetate

Normal butyl acetate and normal butyl acetate-saturated wetting solution shall be used for substances and preparations that cause polyethylene to swell to such an extent that the polyethylene mass is increased by up to 4 % and at the same time causes cracking under stress, in particular for phyto-sanitary products, liquid paints and esters.

Normal butyl acetate of (98–100) % concentration shall be used for preliminary storage in accordance with **7.3**.