Guide
on
Hot Water Bath Testing
and
Its Alternatives

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Foreword

Changes in regulations, progresses in technologies, committed high safety standards and continuous product innovations are demanding up-to-date information as reflected in the relaunch of this excellent guide.

It is my pleasure, as President of the European Aerosol Federation, to introduce to you the second edition of the FEA *Guide on Hot Water Bath Testing and Its Alternatives*.

Since mid of the last century the hot water bath test has maintained its dominant role in testing aerosol products. However line speed, sensitivity of packaging's towards corrosion or sustainability attributes have motivated the industry to strive for alternative solutions to the traditional water bath.

Over the last few years first companies have moved towards the Alternatives and more will certainly follow. To get all parameters and requirements in proper order this guide gives very helpful directions in setting up the company specific conditions to cope with the overall aim to deliver safe products to the market place.

This guide does also connect with the regulatory background as there are the ADR and ADD as the most relevant legal frames in this circumstance.

This Guide is recommended by FEA as a practical contribution to good and safe practice in aerosol testing. Experts from the European aerosol industry and the National Aerosol Associations updated it. This Guide, of course, does not supersede national enforcement of the legislation.

Once more such a guide can only be created and maintained up-to-date with experts among all parties involved, be it can and valve manufacturers or aerosol fillers. Therefore I would like to acknowledge the excellent work the Task Force has done and thank them herewith.

I hope you find the Guide helpful.

Rolf Bayersdörfer
FEA President
October 2016

Contents

Chapter 1

Introduction

Aerosol dispensers (aka. aerosols) have been in commercial production since the late 1940s. Over this period their design, manufacture and filling have undergone substantial development and, with ever increasing consumer demands for aerosols, will continue to do so.

Aerosols use internal pressure generated by a liquefied or compressed gas propellant (or a mixture of the two) to provide the driving force which dispenses the product. This pressure is significant and any failure of the structural integrity of the aerosol could release a considerable amount of destructive energy, thus aerosols are classed as dangerous goods. Further, because many aerosols contain flammable ingredients such as ethanol or hydrocarbon propellants, it is essential to confirm that the aerosol is not leaking before dispatch from the filler. It is therefore a legal requirement to demonstrate that an aerosol is safe for transport and sale. In regulatory terms this means complying with EU and international transport regulations for dangerous goods and the Aerosol Dispensers Directive 75/324/EEC.

Investigations in the 1940s found that, during transport and distribution in hot climates, temperature of pallet loads of aerosols can rise to around 50°C. The hot water bath test was therefore developed to test filled aerosols to ensure that none would burst after leaving the factory. Using this test method every filled aerosol is immersed in a hot water bath for between two and three minutes to allow the contents to equilibrate at the bath temperature. It was subsequently realised that the hot water bath test method can also be used to detect leaking aerosols by monitoring for bubbles of gas escaping from the aerosols whilst they are immersed in the hot water. Studies have shown that the hot water bath test can detect leak rates below levels which do not present a fire risk in transport, distribution or consumer use.

When the hot water bath test was developed, filling lines operated at about 40-60 aerosols per minute, whereas modern manufacturing equipment may fill over 300 aerosols per minute. In order to achieve the required residence time for the aerosols, a modern water bath needs to be up to 10 times larger than when the water bath test was developed fifty years ago. The alternative, to operate the bath at a higher temperature than 50°C, increases the risk and consequences of aerosol failure. With the continual drive to improve manufacturing efficiencies and the use of even faster filling lines, there can be real problems at existing factories to find enough space for the larger water baths.

In the 1990s, FEA investigated possible ‘alternative methods’ identifying a number that were under development. FEA concluded that for an ‘alternative method’ to be acceptable it must be shown to be as effective as the hot water bath test at eliminating faulty and leaking aerosols. In 2002, to demonstrate this principle FEA developed an ‘alternative method’ which consists of an integrated quality assurance and testing package. It is based on the principle that quality assurance and on-line testing may be used to ensure that all sub-standard aerosols are eliminated before they leave the filling line. FEA then validated an ‘alternative method’ by running it prior to and in series with a fully functioning water bath for about a year during which time over 12 million aerosols, made in tinplate, were tested. During the period of the trial no aerosols burst in the hot water bath and just over 100 leaking aerosols were identified by both systems. The work was overseen by an independent safety expert.

In late 2003 FEA presented the work to the UN Sub-Committee of Experts on the Transport of Dangerous Goods which accepted that an ‘alternative method’ is able to provide an equivalent level of safety to the hot water bath test. The report of the trial can be obtained at: www.unece.org/trans/doc/2003/ac10c3/UN-SCETDG-24-inf49e.pdf.

The UN Committee agreed to insert into the UN 2005 Model Regulations for the Transport of Dangerous Goods text which prescribes a framework with criteria for acceptable ‘alternative methods’. The option to use an ‘alternative method’ to ensure that filled aerosols are safe to be transported as ‘dangerous goods in limited quantities’ is implemented in the EU in all modal transport regulations (e.g. ADR[1]) and so into national law in all Member States since 1 July 2007.

The second Adaptation for Technical Progress (ATP) (Directive 2008/47/EC) of the Aerosol Dispensers Directive 75/324/EEC defines criteria for permitted ‘alternative methods’ in line with transport of dangerous goods legislation. Aerosol manufacturers who use an approved ‘alternative to the water bath’ are allowed to apply the reversed epsilon symbol ɜ to the finished aerosol.

Under both the transport regulations and the Aerosol Dispensers Directive, fillers of aerosols are required to subject all filled aerosol dispensers to the hot water bath test or an approved alternative unless specifically derogated. Statistical sampling of aerosols or doing neither are not permitted under the legislation.

The objective of this Guide is to provide advice to ensure aerosols leaving the aerosol filling operation are safe for transport and sale to the consumer. It should be read in conjunction with the ‘FEA Guidelines on Basic Safety Requirements in Aerosol Manufacturing’ which describes how to make the aerosols safely.

[1] The European Agreement Concerning the International Carriage of Dangerous Goods by Road

Chapter 2

The Hot Water Bath Test

The advice set out in this section is designed to ensure that water bath testing of aerosols is carried out safely and complies with EU and international transport regulations and the Aerosol Dispensers Directive.

2.1. The Hot Water Bath Test Regime

The required regime for hot water bath testing of aerosols was initially set out in Clause 6.2.4.3.2.1 of the ADR 2007, (Clause 6.2.6.3.2 of the latest edition of the ADR - see Annex A) and Section 6 of the Annex to the Aerosol Dispensers Directive 75/324/EEC (see Annex B). The wording is slightly different for each regulation, but results in the same test conditions.

<u>All</u> filled aerosols, including compartmented aerosols and those containing compressed gas propellants (for example, carbon dioxide or nitrogen) in place of, or in combination with, liquefied gas propellants must be tested in a hot water bath before being transported. It is recommended that the hot water bath test is carried out immediately after check-weighing (or pressure testing if used) as this will ensure that all faulty aerosols are removed at the earliest opportunity.

2.1.1 The Test Conditions

The temperature of the water in the bath must be at least 55°C (50°C if the liquid phase of the aerosol does not exceed 95% of the capacity of the aerosol at 50°C). Aerosols must be in the water bath long enough for the internal pressure of each aerosol to reach that which would be reached under equilibrium conditions at 55°C (50°C if the liquid phase does not exceed 95% of the capacity of the aerosol at 50°C). It will be necessary to establish by experiment prior to first production the residence time in the hot water bath. For most aerosols this is likely to be between 2 and 3 minutes. Further explanations are provided in standard FEA 606 *Filled aerosol packs – Water bath testing – Verification of conformity with legislation*.

It is important to monitor that the correct temperature of the water in the bath is maintained. Temperature sensors must be positioned in water that is being circulated because the local temperature in a pocket of stagnant water may be significantly different from that of the main body of the water. Similarly if the sensor is placed too near the hot water inlet, temperatures higher than the main body of water and above the desired operating temperature will be recorded. This could lead to a lower bath temperature or unnecessary downtime for the water bath. The temperature should be monitored continuously and regular manual check results recorded and used for trend analysis and maintenance purposes.

Aerosols passing through the water bath must be monitored continuously to check that they are not visibly distorted and/or are not leaking, any such aerosols must be removed as they have failed the test. Monitoring can be done by a number of methods ranging from staff observing the water bath for distorted aerosols and for bubbles from leaking aerosols, to the use of automated leak detectors. A number of devices are available from equipment suppliers. In the case of manual observation, it is general industry practice to relieve observers every 20 minutes, as experience has shown that the incidence of missed leakers rises significantly after this time. It is also recommended that filling lines running at speeds of more than 50-60 cans per minutes should not rely on manual observation to identify leaking or distorted aerosols. Above this line speed, the turbulence caused by the cans moving through the water makes identifying bubbles of leaking propellant extremely difficult. It is important to maintain the clarity of the water in the test bath so that fine leakage bubbles can be detected clearly. It is recommended that regular tests should also be carried out at least once per shift using aerosols modified to produce a range of standard leaks to ensure that they can be detected in the water bath.

Where excessive numbers of aerosols fail the test, the filling line should be stopped and an investigation made into the cause(s) of failure. The filling line should not be restarted until those causes have been rectified.

Although not specified in the transport of dangerous goods legislation, to meet the requirements of the Aerosol Dispensers Directive immersion of the aerosols is necessary. It is recommended total immersion of the aerosol (including the valve stem) because it will ensure that any leakage from the valve assembly or valve crimp as well as the aerosol body can be detected. However total immersion may result in water being trapped in the aerosol valve cup resulting in corrosion and aerosol can failure at a later date. It is therefore important to dry aerosols after water bathing. The use of rust inhibitors in the water bath should also be considered but may need constant replenishment for baths where a water ‘feed and weir’ system is in use. The increased energy use because of the driers or the constant replenishment of the hot water has led some aerosol fillers to place aerosols in the water bath up to but not including the valve cup and combined this with using gas leak detectors placed at or near the exit to the water bath or other means to identify leakages. The aerosols are heated and so the key need here is for the filler to document that, in terms of identifying leaking aerosols, the system in use is equivalent to total immersion.

2.1.2 Heat Sensitive Aerosols

The transport of dangerous goods legislation allows the temperature of the hot water to be reduced to between 20°C and 30°C for aerosols whose contents are heat sensitive, or when the aerosol cans are made of a material which softens at the test temperature (55°C). In these cases one aerosol out of every 2000 should be tested in a water bath at 55°C (50°C if the liquid phase does not exceed 95% of the capacity of the aerosol at 50°C). All aerosols tested at the higher temperature must be rejected as unfit for sale due to the adverse effects of the water temperature. The transport of dangerous goods legislation also exempts certain aerosols from testing in a hot water bath, but the strict conditions for the use of this derogation are not likely to be met by the majority of aerosols (see Chapter 4).

Note: This provision under ADR 6.2.6.3.1.1 is not an alternative to the hot water bath test and is currently not permitted by the Aerosol Dispensers Directive (ADD) 75/324/EEC. However the cold final (alternative) test methods prescribed by the Aerosol Dispensers Directive ADD 75/324/EEC have to be used for heat sensitive aerosols before their placing on the EU market.

2.1.3 Aerosols Containing Non-flammable Compressed Gas Propellants

As mentioned in Section 2.1 above, both ADR and the ADD require that all filled aerosols are tested in a Hot Water Bath unless there is an approved alternative in place. There are no exemptions either for compartmented aerosols or for aerosols using non-flammable compressed gas propellants. Further for non-flammable aerosol formulations where leakage is not a safety issue, the Hot Water Bath Test can be regarded as an essential part of the quality management system.

2.2. Water Bath Safety

Because of the potential hazards involved in water bath testing, it is essential that operatives are fully trained in the operation of the bath and in the procedures to be used if leaking aerosols are detected or an aerosol fails. Proper training in the use of Personal Protective Equipment is crucial because in the hot water bath an aerosol could rupture with considerable force.

2.2.1 Design of the Water Bath

The water bath should be subject to a risk assessment to determine the proper ATEX zone classification.
The water bath should be fully guarded to protect operatives from injury caused by ejected product or aerosols behaving as projectiles. The shock of the first aerosol failing may result in subsequent sympathetic failure of adjacent aerosols. The area immediately above the water bath should have extraction to remove any gases or vapours and to keep the guarding and inspection points free from excess condensation. The extraction system will need to be compatible with the zone classification for the water bath. When using hand-inserted testing baskets in a non-automatic water bath an immersion timer should be used and the basket should have a locking mesh guard hinged on one side.

2.2.2 Control of Water Temperature

The main problem with overheating of the water in the bath is the increased risk of aerosol failure, release of flammable gases or vapours and the failed aerosols behaving as projectiles. This may be achieved well below the boiling point of water and rupture of an aerosol may result in significant local damage to the water bath as well as injuries to personnel.

Experience has shown that all water baths, automatic and non-automatic, should be fitted with an excessive temperature cut-out device in addition to a thermostat. The temperature should be monitored continuously and regular manual check results recorded and used for trend analysis and maintenance purposes. Over-temperature in the water bath should activate an alarm and trigger a device to lift the aerosols out of the bath. An accumulation table or buffer should be fitted downstream from the water bath to allow the bath to discharge its contents fully in the event of a downstream equipment failure.

It should also be noted that temperatures in the water bath or associated pipework are in the region where micro-organisms, in particular *legionella*, can proliferate. Guidance on dealing with this issue can be obtained for example from WHO[2] or National Health and Safety Authorities[3].

2.2.3 Breakdown Procedures

In the event of a downstream equipment failure, aerosols should be prevented from entering the water bath until the failure has been rectified. A method should be in place to remove aerosols from the hot water, options include to continue running the line until all aerosols have exited the bath or preferably a device to lift the aerosols out of the water. One alternative method that could be used for smaller water baths is to remove the hot water by opening the bath drain plug allied with adding cold water either, by increasing the cold water make-up feed to a maximum or using a hose pipe. As the hot water is removed, the operator should monitor the aerosols and reject any substandard ones in the normal way. Aerosols removed from the water bath during a 'shut-down' should be quarantined and re-introduced to the line upstream of the water bath on restart.

2.2.4 Rejected Aerosols

Aerosols rejected by the water bath will be either distorted and could burst or could be leaking. It is therefore important to have safety systems in place that are able to handle these aerosols. A number of methods are available to reject aerosols from the water bath. Soft reject systems should be used especially with aluminium cans because they may produce sparks when striking other metal (particularly rusty) surfaces. The aerosol reject systems and procedures, and the storage areas for rejected aerosols should be subjected to a risk assessment to determine the proper ATEX zone classification. This is because the water bath and its reject bin may contain a flammable atmosphere. Further advice may be obtained from the FEA *Guidelines on Basic Safety Requirements in Aerosol Manufacturing*.

2.3. Waste Water

Water discharged from the water bath for whatever reason should be checked for unacceptable levels of contaminants before being released into the general sewage system. A failure to test water discharged from water baths may result in 'Effluent Consents' being exceeded with the consequent possibility of prosecution or of excess sewage charges. It is recommended that water from the bath is discharged into an effluent holding system where further treatment can be made if necessary.

[2] WHO, Legionella and the prevention of legionellosis, http://www.who.int/water_sanitation_health/emerging/legionella_rel/en/

[3] HSE (UK), Legionella and Legionnaires' disease, http://www.hse.gov.uk/legionnaires/

2.4. Record of Testing

The Aerosol Dispensers Directive requires the marketer to guarantee that all aerosols have been tested according to the requirements set out in the Annex to the Directive. The transport of dangerous goods legislation does not specify how a marketer should prove that aerosols comply with the requirements. However, Clause 6.2.6.4 does specify that the ADR requirements for the construction and testing of aerosol dispensers are met by compliance with the Aerosol Dispensers Directive. For both regulatory regimes, maintaining dossiers of evidence and test records would be a good way to demonstrating compliance with requirements.

Chapter 3

‘Alternative Test Methods’

The parameters for ‘alternative test methods’ are prescribed in ADR 6.2.6.3.2 (see Annex A), they must have four key elements:

- A Quality System that ensures all aerosols that leak or are deformed are rejected and not offered for sale.
- Pressure testing of all empty aerosols at least at two-third of the design pressure of the aerosol container to ensure that they do not deform when filled and leak at a rate less than 3.3×10^{-2}mbar.l.s^{-1}.
- Each filled aerosol dispenser shall be weighed to detect and reject overfilled aerosol dispensers.
- Leak testing of all filled aerosols to detect that they do not leak at a rate greater than 2.0×10^{-3}mbar.l.s^{-1} at 20°C.

The use of an alternative test method is always subject to approval by the national Competent Authorities for the Transport of Dangerous Goods, which are responsible for the enforcement of such provisions.

To take advantage of this option, the company will need to verify that their ‘alternative test method’ provides an equivalent level of safety to the hot water bath test.

This verification can be an extensive task, unless the company chooses to implement an alternative test method that is closely based on the water bath alternative method that has already been used to establish the existing legislative provisions as outlined below.

3.1. The Criteria to establish an Alternative Test Method based on the UN Model Regulations

The alternative test method presented to the UN Sub-Committee of Experts on the Transport of Dangerous Goods by the FEA was an integrated Quality Assurance and testing package which aimed to ensure substandard aerosols are identified and rejected before they enter transport and distribution.

3.1.1 Key Elements

An 'alternative test method' will include the following key elements:

i) Quality Assurance procedures are used to ensure that only cans that are pressure stable and leak-tight are filled. Central to this is that all empty aerosol cans are leak and pressure tested to at least two thirds of the design pressure of the aerosol[4].

ii) Quality Assurance procedures are used to ensure that all valves have all their components in place and will be pressure stable and leak tight once crimped on a can.

iii) Quality Assurance procedures are used during handling to check that only high quality aerosols are produced.

iv) Quality Assurance procedures are used during filling to check that only high quality aerosols are produced. Procedures include:

   a) Checks on the clinching/crimping equipment settings to maintain the correct valve crimp dimensions.
   b) An in-line check-weigher system to ensure overfilled aerosols are rejected.
   c) A micro-leak detector on the filling line to test the valve and valve crimp of all filled aerosols for leaks.

3.1.2 Rejected Aerosols

Filled aerosols rejected by the check weigher or micro-leak detector may be over filled and unstable or else leaking. It is therefore important to have safety systems in place that are able to handle these aerosols. A number of methods are available to reject aerosols from the filling line; soft reject systems that avoid further damage to the aerosols are recommended. The aerosol reject systems, procedures and the storage areas for rejected aerosols should be subjected to a risk assessment to determine the proper ATEX zone classification. This is because this part of the filling line, especially the reject bins, may contain a flammable atmosphere.

Further advice may be obtained from the FEA *Guidelines on Basic Safety Requirements in Aerosol Manufacturing*.

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[4] The 'design pressure' in the ADR provisions corresponds to the 'test pressure' in the ADD and not to the 'rating' of the can.

The 'design pressure' of an aerosol container in the ADR provisions is defined as 50% higher than the maximum internal pressure at 50°C of the filled aerosol product. So, for example, if the maximum internal pressure of an aerosol formulation is 9-bar, then the 'design pressure' of the aerosol container must be 13.5-bar.

In the EU, the aerosol industry defines cans by their 'rating' which is based a test in which a sample can has been filled with water and subjected for 25 seconds to a specified pressure without any leakage or visible or permanent asymmetric or major distortion being caused. This pressure is known as the 'can rating, for example a 15-bar rated can has been tested to a pressure of 15-bar and is suitable for a formulation containing a maximum internal pressure at 50°C of 10 bar.

For the purposes of selecting cans for the 'alternative test method', the rating of the can is not relevant; it is the maximum internal pressure at 50°C that is important and for economic reasons over-specification on the can rating may be necessary. Aerosol can makers will need to clearly communicate to aerosol fillers the maximum internal pressure at 50°C that may use when running an alternative.

Certain aerosol containers could not fit these testing requirements. In such a case those containers shall not be used with the 'alternative method'.

3.2. Verification of an Alternative Test Method

As both ADR and the Aerosol Dispensers Directive only describe criteria for alternative test methods, companies wishing to use an alternative test method to the hot water bath test may use any test system fulfilling those criteria which is approved by their national competent authority. The alternative test method developed by FEA (see Annex C) has already been proven to comply with the requirements of the transport of dangerous goods legislation. Therefore by closely following this example, the verification exercise may be limited to ensuring that the company has implemented and is adhering to this alternative test method.
It is strongly recommended that in any case verification be done by an approved independent third party audit of the whole system. In time, the national Competent Authorities may decide to accept internal audits as evidence of compliance, but this is not likely until experience has been gathered from operating alternative test methods.

The filler must compile and maintain a dossier of evidence to demonstrate that a suitable Quality Assurance system is in place and adhered to. The dossier will need to contain documentation from the can and valve making operations as well as the aerosol filling operation.

Key elements of the dossier will be documentation of:

- The organisational structure and management responsibilities for ensuring adherence to the requirements of the 'Alternative Method'.
- The QA systems for each actor in the supply chain.
- The procedures for handling during the entire process.
- The procedures for pressure and leak testing empty aerosols.
- The procedures for checking the aerosol filling equipment.
- The procedures for check-weighing filled aerosols.
- The procedures for leak testing filled aerosols.
- Appropriate inspection reports, test data, calibration data, and certificates to demonstrate that the procedures are working correctly.
- Records of audits carried out periodically to check that the system is still adequate and efficient and that any critical issues identified are immediately resolved and the system re-audited.

As an example for the practical implementation for an alternative to the water bath test, Annex C sets out details of the key elements of the criteria for alternative test methods developed by FEA and information to be included in the dossier on the Quality Assurance systems and the procedures. The verifier will give particular attention to examining the dossier for evidence that audits are carried out by the can and valve makers and the aerosol filler to ensure that Quality Assurance procedures are in place and being followed correctly.

Once the alternative test method has been verified as compliant with the requirements set out in Section 6.2.6.3.2 of ADR, the national Competent Authorities should be notified. The alternative test method will also need to be audited periodically to ensure that it remains adequate and efficient. This can be done as part of the normal auditing of the Quality Assurance systems, but should be recorded separately. Any proposed changes will need to be notified to the national Competent Authorities before they are implemented.
At the time of the approval, it is recommended that the changes which need to be notified are discussed and clarified with the national Competent Authorities.

3.3. ‘Alternatives’ and Aerosol Dispensers Directive

The Aerosol Dispensers Directive 75/324/EEC permits final ‘alternative’ test methods. Aerosol manufacturers who use an approved alternative to the hot water bath test are allowed to apply the reversed epsilon symbol Ɜ to their finished aerosols.

Chapter 4

Exemptions from Water Bathing

Section 6.2.6.3.3 of ADR (see Annex A) exempts certain pharmaceutical aerosols that are required to be sterile, from the need to be tested in a hot water bath or comply with an approved alternative test method. However, to qualify for this exemption the aerosols must:

- contain pharmaceutical products with non flammable gases;
- be made according to Good Manufacturing Practices (GMPs) as defined by the World Health Organisation (WHO);
- be made to an equivalent level of safety as demonstrated by using alternative methods for leak detection and pressure resistance such as helium detection and water bathing 1 in 2000 aerosols from each bath.

As there are currently no such exemptions under the Aerosol Dispenser Directive, pharmaceutical companies in Europe will have to use an alternative test method rather than this exemptions regime.

Concerning the transport of aerosols, the general exemption from ADR for *gases contained in foodstuffs or beverages* does not apply to food aerosols which fall under the scope of the ADR provisions.
Concerning the placing of aerosols on the European Single Market, food aerosols, as with any other aerosol-type, fall under the scope of the Aerosol Dispensers Directive 75/324/EEC.

Chapter 5

Enforcement

In principle national enforcement of these provisions will not differ, so that all provisions are measured by the same standards at European and international levels. If it differs slightly, contact with the national aerosol associations to get further information on specific requirements is recommended.

Annex A

Extract from ADR

Part 6 **Requirements for the construction and testing of packagings, intermediate bulk containers (IBCs), large packagings, tanks and bulk containers**

6.2.6 **General requirements for aerosol dispensers, small receptacles containing gas (gas cartridges) and fuel cell cartridges containing liquefied flammable gas**

6.2.6.3 **Tightness (leakproofness) test**

Each filled aerosol dispenser or gas cartridge or fuel cell cartridge shall be subjected to a test in a hot water bath in accordance with 6.2.6.3.1 or an approved water bath alternative in accordance with 6.2.6.3.2.

6.2.6.3.1 *Hot water bath test*

6.2.6.3.1.1 The temperature of the water bath and the duration of the test shall be such that the internal pressure reaches that which would be reached at 55°C (50°C if the liquid phase does not exceed 95% of the capacity of the aerosol dispenser or the fuel cell cartridge, gas cartridge at 50°C). If the contents are sensitive to heat or if the aerosol dispensers, gas cartridges or the fuel cell cartridges are made of plastics material which softens at this test temperature, the temperature of the bath shall be set at between 20°C and 30°C but, in addition, one aerosol dispenser in 2000 shall be tested at the higher temperature.

6.2.6.3.1.2 No leakage or permanent deformation of an aerosol dispenser, gas cartridge or the fuel cell cartridge may occur, except that a plastic aerosol dispenser, gas cartridge or the fuel cell cartridge may be deformed through softening provided that it does not leak.

6.2.6.3.2 *Alternative methods*

With the approval of the competent authority alternative methods that provide an equivalent level of safety may be used provided that the requirements of 6.2.6.3.2.1 and, as appropriate, 6.2.6.3.2.2 or 6.2.6.3.2.3 are met.

6.2.6.3.2.1 Quality system

Aerosol dispenser, gas cartridge or the fuel cell cartridge fillers and component manufacturers shall have a quality system. The quality system shall implement procedures to ensure that all aerosol dispensers, gas cartridges or the fuel cell cartridges that leak or that are deformed are rejected and not offered for carriage.

The quality system shall include:

(a) A description of the organizational structure and responsibilities;

(b) The relevant inspection and test, quality control, quality assurance, and process operation instructions that will be used;

(c) Quality records, such as inspection reports, test data, calibration data and certificates;

(d) Management reviews to ensure the effective operation of the quality system;

(e) A process for control of documents and their revision;

(f) A means for control of non-conforming aerosol dispensers, gas cartridges or the fuel cell cartridges;

(g) Training programmes and qualification procedures for relevant personnel; and

(h) Procedures to ensure that there is no damage to the final product.

An initial audit and periodic audits shall be conducted to the satisfaction of the competent authority. These audits shall ensure the approved system is and remains adequate and efficient. Any proposed changes to the approved system shall be notified to the competent authority in advance.

6.2.6.3.2.2 Aerosol dispensers

6.2.6.3.2.2.1 Pressure and leak testing of aerosol dispensers before filling

Each empty aerosol dispenser shall be subjected to a pressure equal to or in excess of the maximum expected in the filled aerosol dispensers at 55 °C (50 °C if the liquid phase does not exceed 95% of the capacity of the receptacle at 50 °C). This shall be at least two-thirds of the design pressure of the aerosol dispenser. If any aerosol dispenser shows evidence of leakage at a rate equal to or greater than 3.3×10^{-2} mbar.l.s^{-1} at the test pressure, distortion or other defect, it shall be rejected.

6.2.6.3.2.2.2 Testing of the aerosol dispensers after filling

Prior to filling the filler shall ensure that the crimping equipment is set appropriately and the specified propellant is used.

Each filled aerosol dispenser shall be weighed and leak tested. The leak detection equipment shall be sufficiently sensitive to detect at least a leak rate of 2.0×10^{-3} $mbar.l.s^{-1}$ at 20 °C.

Any filled aerosol dispenser which shows evidence of leakage, deformation or excessive mass shall be rejected.

6.2.6.3.3 With the approval of the competent authority, aerosols and receptacles, small, are not subject to 6.2.6.3.1 and 6.2.6.3.2, if they are required to be sterile, but may be adversely affected by water bath testing, provided:

(a) They contain a non-flammable gas and either

(i) contain other substances that are consistent parts of pharmaceutical products for medical, veterinary or similar purposes;

(ii) contain other substances used in the production process for pharmaceutical products; or

(iii) are used in medical, veterinary or similar applications;

(b) An equivalent level of safety is achieved by the manufacturer's use of alternative methods for leak detection and pressure resistance, such as helium detection and water bathing a statistical sample of at least 1 in 2000 from each production batch; and

(c) For pharmaceutical products according to (a) (i) and (iii) above, they are manufactured under the authority of a national health administration. If required by the competent authority, the principles of Good Manufacturing Practice (GMP) established by the World Health Organization (WHO)[5] shall be followed.

6.2.6.4 ***Reference to standards***

The requirements of this section are deemed to be met if the following standards are complied with:

- for aerosol dispensers (UN No. 1950 aerosols): Annex to Council Directive 75/324/EEC[6] as amended and applicable at the date of manufacture;

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[5] *WHO Publication: "Quality assurance of pharmaceuticals. A compendium of guidelines and related materials. Volume 2: Good manufacturing practices and inspection".*

[6] *Council Directive 75/324/EEC of 20 May 1975 on the approximation of the laws of the Member States relating to aerosol dispensers, published in the Official Journal of the European Communities No. L147 of 9.06.1975.*

- for UN No. 2037, small receptacles containing gas (gas cartridges) containing UN No. 1965, hydrocarbon gas mixture n.o.s, liquefied: EN 417:2012 Non-refillable metallic gas cartridges for liquefied petroleum gases, with or without a valve, for use with portable appliances - Construction, inspection, testing and marking.

- for UN No. 2037 small receptacles containing gas (gas cartridges) containing non-toxic, non-flammable compressed or liquefied gases: EN 16509:2014 Transportable gas cylinders – Non-refillable, small transportable, steel cylinders of capacities up to and including 120 ml containing compressed or liquefied gases (compact cylinders) – Design, construction, filling and testing (excluding clause 9).

Annex B

Extract from the Annex to Aerosol Dispensers Directive 75/324/EEC

6. TESTS

6.1. Test requirements to be guaranteed by the person responsible for marketing

6.1.4. Final inspection of filled aerosol dispensers

6.1.4.1. Aerosol dispensers shall be subject to one of the following final test methods.

(a) Hot water bath test
Each filled aerosol dispenser shall be immersed in a hot water bath.
   (i) The temperature of the water and the duration of the test shall be such that the internal pressure reaches that, which would be exerted by its contents at a uniform temperature of 50 ºC.
   (ii) Any aerosol dispenser showing visible permanent distortion or a leak must be rejected.

(b) Hot final test methods
Other methods for heating the contents of aerosol dispensers may be used if they guarantee that the pressure and temperature in each filled aerosol dispenser reach the values required for the hot water bath test and distortions and leaks are detected with same precision as in the case of the hot water bath test.

(c) Cold final test methods
An alternative cold final test method may be used if it is in accordance with the provisions of an alternative method to the hot water bath test for aerosol dispensers specified in point 6.2.4.3.2.2 of Annex A to Directive 94/55/EC.

6.1.4.2. For aerosol dispensers the contents of which undergo a physical or chemical transformation changing their pressure characteristics after filling and before first use, cold final test methods according to point 6.1.4.1(c) should be applied.

6.1.4.3. In case of test methods according to points 6.1.4.1 (b) and 6.1.4.1 (c):

(a) The test method must be approved by a competent authority.
(b) The person responsible for the marketing of aerosol dispensers must submit an application for approval to a competent authority. The application must be accompanied by the technical file describing the method.
(c) The person responsible for the marketing of aerosol dispensers must, for surveillance purposes, keep the approval of the competent authority, the technical file describing the method and, if applicable, control reports readily available at the address specified on the label in accordance with point (a) of Article 8(1).
(d) The technical file must be established in an official Community language or a certified copy thereof must be available.
(e) ‘competent authority’ means the authority designated in each Member State under Directive 94/55/EC.

6.2. Examples of inspection tests which may be carried out by Member States

6.2.2. *Tests on filled aerosol dispensers*

Air and water-tightness inspection tests shall be carried out by immersing a representative number of filled aerosol dispensers in a bath of water. The temperature of the bath and the period of immersion must be such as to enable the contents of the aerosol dispenser to attain a uniform temperature of 50°C during the time required to ensure that there is no bursting or rupture.

Any batch of aerosol dispensers which does not pass these tests must be considered unsuitable for use.

Annex C

Example for the practical implementation of an Alternative to the Hot Water Bath Test as developed by FEA

Any alternative test method has to be equivalent to the current hot water bath test.
To this end an alternative test method must satisfy the following conditions:

a) Be safe to operate and adhere to the safety legislation in the country where it is to be operated. It may only be used with the agreement of the appropriate Competent Authority.
b) Be validated by comparison with the traditional hot water bath test. It must not allow any aerosols to pass that subsequently burst or deform in a water bath that is operating under its normal conditions.
c) Be shown to be as effective at identifying leaking aerosols as the hot water bath test.

It is strongly recommended that an independent assessor verifies any alternative test method.

The alternative test method developed by FEA includes an integrated Quality Assurance and testing package involving the can and valve manufacturers as well as the aerosol filler.

Quality Assurance System

Can and valve makers and aerosol fillers must all have documented Quality Assurance systems in place that are functioning correctly. The Quality Assurance systems should contain the following elements:

- All quality documents must be issued under the authority of the Quality Assurance Manager. There must be stringent document control which assures that procedures are in accordance with the correct document revision and unauthorised changes to procedures do not occur.
- Minor corrective actions carried out by the production team under appropriate supervision must be recorded. Production management should review minor corrective actions.
- Major corrective actions must be authorised and recorded by the production management.
- Quality records must be legible.
- All production quality documentation must be collated and retained for at least five years.

- All test equipment that is critical to quality must be identified and routinely calibrated. For example, the measurement apparatus for determining the crimp dimensions and the dip tube length should each be identified with a unique number, and after calibration a label attached to them that shows the date when its next calibration is due. The operator can therefore carry out inspections and measurement with test equipment known to have been calibrated and not to have exceeded its next calibration date.
- The filler must carry out periodic inspections of suppliers for compliance with agreed quality procedures.
- The filler must receive the supplier certification for the components before they are released to production.
- Components must be identifiable so that they can be traced back to the manufacturer's batch number. From this identification, it should be possible for the production run of the can/valve to be identified and production record sheets to be retrieved.
- The Filler must be able to identify from the filled aerosol the production runs of the can and valve components. Each aerosol must bear a code from which it is possible to know when and where it was filled. The filler should be able to relate this code to the can and valve manufacturers' batch numbers thus providing full traceability to the production runs of the cans and valves.
- During assembly, in-process inspection and testing must be in place to ensure adherence to the product specifications.
- If too many units are ejected from the filling line as non-conforming, then production must be stopped and the batch quarantined.
- Quarantined stock must be inspected by management to decide if it is satisfactory and what corrective action is required.
- Leaking or overfilled aerosols must be disposed of appropriately.
- The final product must undergo a final inspection and testing protocol.
- Handling procedures must be in place to ensure that there is no damage to final product.
- Product for dispatch must be adequately packaged so as to prevent damage while in transit.
- Transport packages must be adequately labelled in compliance with transport regulations.
- Staff must be trained in relevant procedures and their training recorded.

Testing of Aerosols

<u>Testing of Aerosols</u>

- <u>Empty Aerosols</u>[7]

  Each empty aerosol must be pressure tested to a pressure that is two-thirds of the deformation pressure of the can. If a leak occurs, the empty aerosol must be rejected. For the test to be meaningful, it is critical for the empty aerosol to be supported at the neck end only. If the empty aerosol is supported at the bottom and top then the additional support will strengthen the can and the pressure test may not reveal faults. A functionality test must be carried out to ensure it will detect a leaking can. The performance of the test unit must be continuously monitored to ensure it is not acting erroneously. The required performance of the can pressure test unit is to detect a leak rate equal to or greater than 3.3×10^{-2} mbar.l.s^{-1} at the test pressure.

[7] Certain aerosol containers could not fit these testing requirements. In such a case those containers shall not be used with the 'alternative method'.

Testing of empty aerosols will normally be carried out by the can maker who will need to document the results of the testing as part of the Quality Assurance system.

- Filled Aerosols.
  The aerosol filling line should have the following key elements:
  - Valve Crimper. At the start of a product changeover that requires the crimping head to be reset, the integrity of the new crimp setting should be proven by placing the aerosol in a laboratory hot water bath.
  - Check Weigher. Each filled aerosol should be weighed to check that it is not over filled; for a liquefied gas this will also eliminate any aerosols that are over-pressurised. A system should be in place to automatically reject any over filled aerosols from the filling line into a waste container.
  - Valve and crimp leak detector. Each filled aerosol should be placed in a head that covers the whole valve and will detect a leak from the valve crimp or the valve itself. A functionality test must be carried out on the unit prior to each test to ensure it will detect a leaking can. The performance of the test unit must be continuously monitored to ensure it is not acting erroneously. The required performance is to detect a leak rate of equal or greater than 2.0 x 10^{-3}mbar.l.s^{-1} at the 20°C. A system should be in place to reject any leaking aerosols automatically from the filling line into a waste container.

The Dossier of Evidence

In order to provide proof that the system in place complies with the criteria of an alternative method laid down in the UN Model Regulations and thus meets the requirements of the current ADR Regulations and the Aerosol Dispensers Directive 75/324/EEC, and provides an equivalent level of safety to the hot water bath test it is key that the filler compiles and maintains a dossier of evidence.

The dossier must contain:

- The organisational structure and management responsibilities for ensuring adherence to the requirements of the alternative test method.
- Information to document that the Quality Assurance systems contain all of the elements described above.
- A quality manual that describes how the company carries out internal audits to verify adherence to its systems.
- Inspection records, test and calibration data, test certificates and training record certificates to demonstrate that the procedures are working correctly.
- Inspection reports, test data, calibration data, and certificates etc. to show that the test and calibration regimes for empty and filled aerosol cans comply with the test conditions described above.
- The filler’s dossier must include information that will enable a can or valve to be traced back to its manufacturing run.