8.11 **AEROSOL DISPENSERS (AE)**

# Introduction

Not all characteristics which define the acceptability of aerosol dispensers are incorporated in the specification guideline given below.

The flammability and ignition distance of the spray produced by the dispenser and formulation are of potentially great importance to the user but they must be dealt with by appropriate labelling of the dispensers.

The spray droplet size distribution is relevant to operator risk and may influence efficacy. At present, the measurement is complex and interpretation of the results is not straightforward, and therefore no clause is included.

Aerosol dispensers are expected to withstand corrosion for a minimum of 2 years from the date of release by the manufacturer. Ideally, specifications would include a clause to define the corrosion resistance of the dispenser. At present, no practical method is available to predict whether or not the structural integrity of dispenser could be compromised within 2 years and therefore a clause cannot be included. Manufacturers, users of aerosols and/or other interested parties are invited to develop and propose simple methods based on standard conditions, so that this important omission can be rectified.

Note for preparation of draft specifications. Do not omit clauses or insert additional clauses, nor insert limits that are more lax than those than given in the guidelines, without referring to section 4. If two or more active ingredients are co-formulated, they should have separate specifications. From the “Notes” provided at the end of this guideline, incorporate only those which are applicable to the particular specification.

**...... [ISO common name] AEROSOL DISPENSERS**

[CIPAC number]/AE (month & year of publication)

The material, sampled from any part of the consignment in accordance with the procedure described in Note 1 or any other acceptable procedure, shall comply with the specification.

8.11.1 **Description**

The product shall consist of a liquid ‡formulation in a pressurised, non‑refillable aerosol dispenser, containing propellant(s), synergist(s) and other formulants (see note 2, for restrictions on solvents and propellants), as required, intended for release of the active ingredient into the air in the form of an aerosol. The technical (ISO common name(s)), in the form(s) of …… (see Section 4.2), used in the manufacture of the formulation shall comply with the requirements of WHO specification(s) ......

8.11.2 **Active ingredient and synergist**

8.11.2.1 **Active ingredient identity tests** (Notes 3 and 4)

 The active ingredient shall comply with an identity test and, where an identity remains in doubt, it shall comply with at least one additional test.

8.11.2.2 **[Active ingredient ISO common name] content** (Notes 3 and 4)

 The ...... [ISO common name] content shall be declared (g/kg) and, when determined, the average content measured shall not differ from that declared by more than the appropriate tolerances, given in the table of tolerances, Section 4.3.2.

8.11.2.3 **[Synergist ISO common name] content** (Notes 4, 5 and 6), if required

 The ...... [ISO common name] content shall be declared (g/kg) and, when determined, the average content measured shall not differ from those declared by more than the appropriate tolerances, given in the table of tolerances, Section 4.3.2.

8.11.3 **Relevant impurities**

8.11.3.1 **By-products of manufacture or storage** (Notes 4 and 7), if required

 Maximum: ..….% of the …… [ISO common name] content found under 8.11.2.2.

8.11.4 **Physical properties**

8.11.4.1 **Net content of formulation**

 The minimum net content shall be declared (kg) and, when determined, the average net content shall not be lower than that declared.

8.11.4.2 **Internal pressure** (Note 8 and Figure 1)

 The maximum rated pressure of the dispenser shall be declared on the label and, when measured at 30 ± 2 ºC, the internal pressure shall not exceed ......% of the maximum rated pressure.

8.11.4.3 **Discharge rate** (Note 9)

 The discharge rate of the filled dispenser shall be within the range .….. to …… g formulation/sec.

8.11.4.4 **pH range** (applicable to water-based formulations only) (Note 10)

 pH range: ...... to ......

8.11.4.5 **Clogging of aerosol dispenser valves**

 No clogging shall occur when the aerosol dispenser valves are tested in accordance with the procedure as described in Note 11 or any other acceptable method.

8.11.5 **Storage stability**

8.11.5.1 **Stability at elevated temperature**

 After storage at 54 ± 2 ºC for 14 days (Note 12), the determined average active ingredient content must not be lower than ......% relative to the determined average content found before storage (Note 13) and the formulation shall continue to comply with the clause for:

- by-products of manufacture or storage (8.11.3.1),

and

- the combined weight of the container and contents shall not be less than …% of the original weight.

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Note 1 Sampling

 *General requirements*

a) Samples shall be stored in such a manner that there is no deterioration of the material.

b) The sampling instrument shall be clean and dry.

c) Samples shall be protected against contamination.

 *Sampling, testing and acceptance*

a) In any consignment, all the master cartons containing containers of the same type shall constitute a lot.

b) Samples shall be drawn from each lot and individually tested to ascertain whether the material complies with the specified requirements.

c) Any sample failing to comply with the specified requirements shall be termed as defective. The acceptance number shall be the maximum number of defective samples permissible for a lot to be accepted.

d) The number of containers to be drawn from the lot and the acceptance number shall be as shown in the following Table.

|  |  |  |
| --- | --- | --- |
| Total number of containers in lot | Number of containers to be tested | Acceptance number |
| 300 or less301 to 12001201 to 20002001 to 70007001 to 1500015001 to 2400024001 to 41000over 41000 | 361321294884126 | 012346913 |

e) Each of the containers to be tested shall be drawn from a different master carton which shall be selected at random. In order to ensure randomness of selection, random number tables shall be used. If such tables are not available, the following procedure may be adopted.

 Starting from any master carton, count the master cartons as 1, 2, 3...... r in a systematic manner. Every rth carton shall be drawn, r being the integral part of N/n, where N is the total number of master cartons in the lot and n the number of master cartons to be selected.

Note 2 Solvents not permitted for use in aerosols:

 benzene

 2‑butoxyethanol (ethylene glycol monobutyl ether)

 2‑butoxyethylacetate (ethylene glycol monobutyl ether acetate)

 carbon tetrachloride

 chlorobenzene

 chloroform

 1,2‑dichloroethane (ethylene dichloride)

 2‑ethoxyethanol (ethylene glycol monoethyl ether)

 2‑ethoxyethylacetate (ethylene glycol monoethyl ether acetate)

 n‑hexane

 2‑hexanone (methyl n‑butyl ketone)

 2‑methoxyethanol (ethylene glycol monomethyl ether)

 2‑methoxyethylacetate (ethylene glycol monomethyl ether acetate)

 tetrachloroethylene

 trichloroethylene.

 Propellants. The Montreal Protocol and EU[[1]](#footnote-1)1 directive on the withdrawal of chlorofluorocarbons (CFCs) from aerosols were noted. Hydrocarbon propellants are recommended for insecticide aerosols, provided international safety standards are met by the aerosol producer. Industry should be encouraged to develop alternative and safer propellants and delivery systems.

Note 3 Method(s) of analysis must be CIPAC or AOAC. If the methods have not yet been published then full details, with appropriate method validation data, must be submitted to WHO by the proposer.

Note 4 To remove the contents of the dispenser it should be weighed (to ± 0.01 g, at room temperature so that it is dry on the outside) and either immersed in dry ice (solid carbon dioxide) for a minimum of 1 h or placed in deep freeze (-18 ºC or below) overnight. The container should then be removed to a fume hood and, using a suitable shield, the container should be punctured towards the top with a sharp implement, making a hole or holes that will be sufficiently large for the subsequent introduction and removal of extraction solvent. Allow the propellent to evaporate by allowing the contents to rise to room temperature without applying additional warming. Using a suitable solvent (appropriate to the active ingredient and synergist), thoroughly rinse the contents of the dispenser into a volumetric flask, make to volume with the solvent. If possible, use a solvent that will not remove paint or other external coatings. Using a suitable analytical method, determine the mass of active ingredient and synergist in the rinsate. Dry the rinsed dispenser and re-weigh it to determine the mass of formulation it contained. Use this value to express the content of active ingredient and synergist on a g/kg basis.

Note 5 If the identity of the synergist is considered confidential, the JMPS will consider the requirements for this clause on a case-by-case basis.

Note 6 The method(s) of analysis must be peer validated or CIPAC or AOAC.

Note 7 This clause should include only relevant impurities and the title should be changed to reflect the name of the relevant impurity. The method(s) of analysis must be peer validated.

Note 8 Determination of pressure in finished aerosol packs[[2]](#footnote-2)2.

 *Introduction*

The determination of the pressure existing in the finished aerosol packs is necessary to verify that the true pressure is compatible with the pressure limitations of the pack, and in accordance with the regulations in force.

True pressure is the relative pressure given by an accurate manometer, at a given temperature.

*Objective*

The determination of the true pressure in the finished aerosol pack:

1. in such a way that the measurement affects as little as possible the value of the real pressure;
2. in such a way that the manometer will not be polluted by the product under pressure present in the pack.

*Scope*

The method is recommended for determination of the true pressure of all filled aerosol packs.

*Apparatus*

The following are required:

1. a source of reference gas (nitrogen for instance) from which the pressure can be regulated by means of a control valve;
2. a manometer of high accuracy, if possible cushioned by an oil-bath and adapted to fit the aerosol container on which the measurement will be effected (Figure 1).

The apparatus must be assembled in such a way that, in the state of rest, the manometer is connected to the reference gas (the pressure of this gas being slightly higher than the actual pressure in the pack) and, for taking the measurement, the manometer is connected to the interior of the pack to show the actual pressure.

*Working operation*

1. Make sure that the aerosol dispenser is thoroughly equilibrated to 30 ± 2 ºC.
2. The measuring apparatus must be fitted with an appropriate adaptor for the valve employed.
3. The pressure of the reference gas must be regulated to a value slightly higher than the anticipated pressure of the dispenser.
4. Apply the measuring apparatus to the valve and press lightly in order to open the valve and the slide of the apparatus.
5. Read the true pressure on the manometer, when the needle has stabilized.

*Accuracy of measurement*

The measurement of the true pressure will be the more accurate:

1. with larger aerosol dispenser sizes;
2. with only a small difference in pressure between the reference gas and the true pressure of the dispenser (if required, measurements can be made on additional dispensers after adjusting the reference gas pressure to a value very close to the true pressure);
3. if the dead volume of the manometer is small (less than 2 ml).

*Test report*

The test report must indicate, in addition to the results and test conditions, any relevant working details not specified in the method, especially if they are suspected of having influenced the results.

*Notes*

5.1 It is necessary to recalibrate the manometer frequently, for example with the aid of a manometric balance.

5.2 A non-return valve can be inserted in the apparatus to avoid the aerosol product penetrating the manometer if the pressure of the reference gas is inadvertently much lower than the true pressure in the dispenser.

Note 9 Evaluation of discharge rate of filled aerosol dispensers[[3]](#footnote-3)1

 *Scope*

The method is applicable to the majority of aerosols marketed at present. It may be used, with discretion, for dispenser fitted with vapour-phase taps where there will be a continually changing composition as the contents are discharged. It is not suitable for use in the inverted position for dispensers with vapour phase taps, as there will be a erratic discharge dependent upon the liquid content of the dip tube. Where dispensers are intended to be used in an inverted position, it is vital that the test shall be done in that way. It is important that discharge tests follow the instructions for use given on the dispenser. The method is not intended for use with metering valves.

 *Principle*

The discharge rate of an aerosol dispenser is determined by measuring the quantity of material expelled through the valve in the given time. The exact duration of discharge, normally 10 sec, and the temperature of the dispenser must be carefully controlled for good reproducibility. Normally the test is repeated three times to give three determinations but, in the case of products filled with vapour phase tap valves, it is preferable to reduce the discharge interval to 5 sec and the number of determinations to two. This is to minimise the variation in composition that will occur as the contents are sprayed off. There will be some loss of accuracy using the shorter duration of discharge.

Alternatively, the discharge rate may be determined at different stages in the emptying of the dispenser (e.g. 90%, 70%, 50%, 30% and 10% fill) which then allows the values to be plotted graphically. Where the composition changes with the emptying of the packs, this offers a particularly convenient way of showing the effect of this change. In the case of storage test samples, a single test is normally performed at each examination to conserve the contents.

*Apparatus*

A water bath at 25 °C ± 0.5 °C, a stop watch accurate to 0.2 sec, a balance weighing to 0.1 g and a pressure gauge accurate to 2 psig (0.1 kg.cm-2) are required.

*Procedure*

1. The valve of the aerosol dispenser shall be handled according to label instructions and the valve shall be operated for five sec to remove material in the dip tube which may not be homogenous with the bulk of the filling.
2. The aerosol dispenser shall be immersed in a water bath maintained at 25°C ± 0.5 °C for half an hour, or for sufficiently long for the contents to attain the temperature of the water bath.
3. The aerosol dispenser shall be removed from the water bath, wiped completely dry, the valve operated for one second to remove any water in the valve, the internal pressures of the dispenser shall be measured and the dispenser shall be weighed to within 0.1g.
4. The aerosol dispenser shall then be shaken for 3 sec by hand or other suitable means and the valve shall be operated fully open for 10 sec, timed by the stop watch. During the discharge, the dispenser shall be positioned as indicated in the instructions for use.
5. The aerosol dispenser shall be wiped clean of any liquid and re-weighed to within 0.1 g.

Procedures (b) to (d) shall be repeated twice or more and the internal pressure of the dispensers shall be measured again.

*Calculation*

The difference in weights derived from procedure (e) and procedure (c) shall be divided by 10 (sec). Results should not differ by more than 0.1 g from the mean of the three results. If a greater difference is found at least two more readings should be taken.

*Reporting*

Report valve discharge rates as g.sec-1 at the measured mean pressure of the dispenser. The method, e.g. 3 x 10 sec, 2 x 5 sec or 1 x 5 sec (90%....50%....10%) spraying, etc., shall be quoted.

*Notes*

1. Where discharge rates are to be determined at various stages of pack emptying it is advisable to allow the pack to reach equilibrium at 25 °C in the water bath, shake and spray for 5 sec, re-shake vigorously and re-spray for another 5 sec and replace in water bath. This procedure may then be repeated until the correct pack content is reached. This rather tedious procedure avoids errors arising from the fall in temperature of the contents of the pack during prolonged spraying.
2. Results may show variations from sample to sample that are greater than might be expected from normal manufacturing tolerances. This may be due variations in the valve orifice diameter.
3. The discharge rate of freshly prepared aerosol dispensers will not normally be the same as for samples allowed to “mature”, due to the effect of solvents on the gasket.

Note 10 The pH may be determined by any acceptable method.

Note 11 Testing of valves of filled aerosol dispensers for clogging

 *Apparatus*

a) Fume hood

b) Protective clothing and mask.

 *Procedure*

 Shake the aerosol dispensers thoroughly and, keeping them in an upright position, disperse the contents of each into the fume hood. Actuate the valve in a series of cycles (30 sec on, 30 sec off) until the dispenser is emptied. Examine the valves for clogging.

Note 12 Unless other temperatures and/or times are specified. Refer to Section 4.6.2 of this Manual for alternative storage conditions.

Note 13 Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after the test in order to reduce the analytical error.

Figure 1. Manometric measurement of internal pressure.

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1. 1 European Union. [↑](#footnote-ref-1)
2. 2 European Aerosol Federation, (49 Square Marie-Louise, 1000 Brussels, Belgium). [↑](#footnote-ref-2)
3. 1 European Aerosol Federation 49 Square Marie-Louise, 1000 Brussels, Belgium). [↑](#footnote-ref-3)