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Standard Practice for Storage Testing of Aerosol Products[1]

This standard is issued under the fixed designation D3090; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the storage testing of aerosol products.

1.2 There are two major types of storage tests that may be performed on aerosol products:

1.2.1 *Live Storage Tests,* where the valves are actuated and the determinations are made at relatively frequent intervals (the purpose being to simulate consumer use of aerosol dispensers), and

1.2.2 *Dead Storage Tests,* performed to simulate warehouse storage conditions when shelf-life information is sought.

1.3 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see Section 4.

2. Significance and Use

2.1 Aerosol products are subjected to storage tests to ascertain the shelf-life of the complete package, and to evaluate the degree of suitability of the valve and container components for their intended uses.

2.2 It is impractical to promulgate a standard procedure for conducting storage tests, since variations will be necessitated by differences in the ultimate objective (for example, the primary interest of one test may be concerned with container suitability or shelf-life of a new product in an existing package, while another test may be concerned with valve evaluation).

2.3 It follows that storage testing must be flexible enough to accommodate the small procedural changes required. Thus, this recommended practice will only set forth those principles to be observed in establishing a definite procedure, in order to allow the individual operator the prerogative of adapting these to satisfy his particular requirements.

3. General Requirements

3.1 Before making any aerosol storage tests, the following should be borne in mind:

3.1.1 Sufficient test specimens should be available to replace any that fail during the test, and to make it possible to extend the storage period if desired.

3.1.2 The test schedule and procedure should be well planned. Only if this is followed, can there be any assurance that important developments have not been missed, and that the results will correlate with other storage test results.

3.1.3 The tests should be performed by competent personnel well qualified in the field. Since most of the data is not obtained by direct measurement and is therefore not entirely objective in nature, it is highly desirable to have the same operator perform all of the tests on a given specimen. This, in addition to 3.1.2, will do much to minimize the effect of the human element.

3.2 Before any specimens are committed to storage, the following should apply:

3.2.1 All pertinent background information concerning the problem should be assembled, so that the test specimen can be intelligently set up.

3.2.2 Tests should be conducted to eliminate defective containers and valves (the frequency of such defects should be recorded). To make this segregation possible, pressure determinations, hot bath, vial leakage, and spray tests should be made on each filled dispenser.

3.2.3 Conditions of filling and handling should as closely as possible approximate those that would be encountered commercially.

4. Safety Precautions

4.1 Aerosol storage tests involve a container, valve, or product of unknown compatibility and performance. For this reason, serious accidents could occur. The operator should employ gloves, safety shield, safety glasses, and apparatus with proper controls.

4.2 If, during a test, container perforations or signs of advanced corrosion are found, or if the product, dispensers, or

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valves otherwise become unmerchantable, the entire lot of specimens should be destroyed. Continued testing would waste time and space, and could result in a serious accident.

5. Live Storage Test

5.1 *Test Temperature*—Specimens should be stored at room temperature. In addition, a higher-temperature storage (for example, 98°F (36°C) may be employed. The use of the higher-temperature storage is particularly desirable when a new valve or product is being evaluated. The use of storage temperatures below 32°F (±0°C), or the alternate exposure to subfreezing and elevated temperatures, has considerable merit in the screening of new valves or new valve materials.

5.2 *Test Position and Number of Specimens*—If the purpose of the test is to evaluate a valve, half of the specimens at each storage temperature should be kept in an inverted position. If the product, or any constituent thereof, exerts a detrimental effect on the sealing material of the valve, the conditions may be more readily observed in the case of inverted cans. Six cans inverted and six cans upright for each temperature is the minimum number of specimens for each variable that should be considered. If the test involves only one temperature, ten to twelve cans per variable (upright and inverted) is a more desirable size.

5.3 *Test Time*—The tests are usually considered completed when 10 g or less of formulation remains in the containers. Extension of the tests beyond this point may cause erratic and unreliable results.

5.4 *Examination Schedule*—Examinations of the specimens should be made weekly, or more often if the completion of the test in less total elapsed time is necessary.

5.5 *Failure*—If a valve becomes totally inoperative or fails to operate properly, the container and valve should be immediately torn down to ascertain the cause of failure.

5.6 *Final Examination*—Each container and valve should be critically examined as soon as possible after the final valve actuation of the test.

6. Dead Storage Test

6.1 *Test Temperature:*

6.1.1 Specimens are usually stored at 98°F (36°C) and room temperature, while other temperatures are employed in special cases.

6.1.2 Temperatures from 95 to 100°F (35 to 37°C) (often referred to as incubation temperature) may accelerate container corrosion and leakage, if the containers are so predisposed. However, incubated storage should always be used in conjunction with room temperature since it is often difficult, if not impossible, to predict a normal shelf-life on the basis of 98°F tests alone.

6.1.3 Storage below freezing (0 to 32°F (−17 to 0°C)) is valuable for evaluating the sealing efficiency and suitability of the gasket materials in aerosol valves.

6.1.4 Storage at 130°F (54°C) should be employed when the resistance of the container to structural fatigue is to be determined.

6.2 *Test Position and Number of Specimens:*

6.2.1 Specimens at each storage temperature should be held in both upright and inverted positions.

6.2.2 Enough specimens should be provided for each test so that a minimum of two dispensers of each variable from each temperature can be evaluated and torn down at each scheduled examination. The other specimens remain untouched, with the exception of weighing, until they are needed at a subsequent examination.

6.2.3 A minimum of twelve extra specimens per variable should be stored at each temperature to allow for extension of the test, if this later becomes necessary, and to allow a larger number of specimens to be inspected at the final examination.

6.2.4 Thus, the minimum suggested number of specimens per product, container, or valve variable is as follows:

$$4np(4+y) \tag{1}$$

where:

y = duration of the test, years,
n = number of storage temperatures, and
p = number of storage positions to be employed.

6.2.5 It is usually desirable to include a glass bottle of the aerosol concentrate with the specimens, in order to determine deteriorations that may take place that are independent of the aerosol container. One or two glass aerosol containers may also be included.

6.2.6 It is often useful to place one or two empty metal containers with the specimens, especially for internally lined variables, for possible future references.

6.3 *Test Time*—Most dead storage tests are concluded after 24 months of storage, but a test may be extended for a much longer period, if the previous results and the objective so require.

6.4 *Examination Schedule*—Examinations are usually made at 1, 3, and 6-month intervals, and at 6-month intervals thereafter, until the test is completed.

7. Examination

7.1 The examination of the specimen may be divided into performance determination, container and valve inspection, and product evaluation.

7.2 The performance of the complete specimen may be ascertained by making mass loss, discharge rate (10 s), pressure, and possibly particle size determinations. At each examination, the mass loss and discharge rate should be measured. Internal pressure determinations at each examination are usually not necessary, but it is recommended that the pressures be taken initially and two or three equally spaced times during the test.

7.3 As a check on filling, the volatile-nonvolatile ratio may be determined following the examinations.

7.4 After expulsion of the propellant, the product should be transferred and the container and valve carefully torn down and examined. The metal valve parts should be carefully inspected for evidences of corrosion, and the rubber or plastic components should be checked for swelling, softening, or disintegration. Conditions in the container interiors should then be noted with special emphasis on any staining, detinning, rusting,

pitting, or other indications of corrosion that may be present. Microscopical examination of the valve and container components is recommended, for without this assistance important and indicative developments may be overlooked.

7.5 The product from the containers should be examined for color change and precipitate or sludge formation. If corrosion is found or suspected in the container, it is suggested that the product be analyzed for the moisture, iron, and tin content.

7.6 If any abnormal or undesirable conditions are found in the performance of the valve or product, sufficient additional specimens of the same lot should be examined to confirm the findings.

7.7 If entomological data are required, they may be obtained by using pertinent existing standard procedures.

7.8 See Table 1 for a sample aerosol product storage test (both dead and live storage).

7.9 See Appendix X1 for other tests that may be run in conjunction with an aerosol storage testing program.

8. Keywords

8.1 aerosol products; aerosol products storage; shelf-life of aerosol packaging; storage testing ; storage testing aerosol products

TABLE 1 Sample Aerosol Product Storage Test

	Live Storage	Dead Storage
Product, container; valve variables	2 (product)	2 (product)
Storage temperatures	room temperature, 98°F (36°C)	30°F (–1°C), room temperature, 98°F (36°C), 130°F (54°C)
Storage positions	upright and inverted	upright and inverted
Number of filled cans per formulation	24 (half inverted at each temperature)	144 (half inverted at each temperature)
Total Number of filled cans	48 (half inverted)	288
Duration of test	until completed	2 years
Examination schedule	weekly	1, 3, 6, 12, 18, 24 months
No. of containers examined each examination	48 cans	24 cans (2 per product per temperature per position), all remaining dispensers examined after 24 months shortage (all dispensers weighed)
Examination procedure	mass loss	mass loss
	pressure	pressure
	discharge rate (10 s)	discharge rate (10 s)
	(particle size)	valve examination
	valve and container inspection (at final examination or when failure occurs)	container examination
		product examination

APPENDIX

(Nonmandatory Information)

X1. OTHER RECOMMENDED TESTS

X1.1 There are several other tests that should be considered in conjunction with an aerosol storage testing program.

X1.2 In evaluating a valve for a given formulation, it may be desirable to subject the containers to a continuous discharge test, whereby the containers are emptied in a single burst or a series of long bursts. This procedure is very rapid, and may give valuable clues on the suitability of the valve gasket material for the product. If indications of weakening, disintegration, or undue swelling of the valve gaskets are found by means of this test, particular attention should be directed to the results of the live storage test.

X1.3 Before commercial packaging of a new product or the use of a new container or valve is approved, it is desirable to obtain certain information outside the scope of the laboratory test:

X1.3.1 Filled containers should be subjected to normal handling, cartoning, and shipping operations to determine the suitability of protective devices and the resistance of the containers and valves to shock.

X1.3.2 Containers should be shipped to and stored in warehouses having the extremes in temperatures that could be encountered in the distribution and marketing of the product. After a predetermined storage period in the various locations, the containers should be returned to the laboratory for a complete examination.

X1.4 Once commercial packaging of a product is initiated, a program may be inaugurated whereby defective or complaint dispensers encountered in the field are returned to the laboratory. By this means, a continuous check on the quality is maintained, and it may enable the manufacturer and packer to be forwarned of any difficulty before it becomes serious, so that corrective measures may be taken.