



Designation: F 798 – 97 (Reapproved 2002)

## Standard Practice for Determining Gettering Rate, Sorption Capacity, and Gas Content of Nonevaporable Getters in the Molecular Flow Region<sup>1</sup>

This standard is issued under the fixed designation F 798; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice describes techniques for determining getting rates, sorption capacity, and gas content of nonevaporable getters in the molecular flow region.

1.2 Procedures for activating getters and for determining gas evolution rates are also given.

1.3 The various tests described are mostly destructive in nature. In general, the tests are semiquantitative, but they can be expected to yield comparative information on a single laboratory basis. Multilaboratory reproducibility can be established only with round-robin testing. Single laboratory precision is  $\pm 15\%$  for getting rate and sorption capacity. Multilaboratory reproducibility is estimated at  $\pm 50\%$ . Gas content measurements may have a substantially greater error due to the uncertainty of the temperature.

1.4 Adverse getter-device interactions such as contamination and poisoning can occur. Such problems are beyond the scope of this practice. The user and seller should establish criteria for controlling problems such as chemical reactions, loose particles, getter location, etc.

1.5 *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.* Specific hazard statements are given in Section 4.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

E 296 Practice for Ionization Gage Application to Space Simulators<sup>2</sup>

E 297 Test Method for Calibrating Ionization Vacuum Gage Tubes<sup>3</sup>

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.03 on Metallic Materials. Current edition approved Dec. 10, 2002. Published May 2003. Originally approved in 1982. Last previous edition approved in 1997 as F 798 – 97.

<sup>2</sup> *Annual Book of ASTM Standards*, Vol 15.03.

<sup>3</sup> Discontinued. See 1983 *Annual Book of ASTM Standards*, Vol 15.03.

#### 2.2 American Vacuum Society Standards:

Recommended Practice 2.3 Procedure for Calibrating Gas Analyzers of the Mass Spectrometer Type<sup>4</sup>

Recommended Practices 6.2, 6.4, and 6.5 Procedures for Calibrating Pressure Gages and Their Controls<sup>4</sup>

### 3. Terminology

#### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *nonevaporable getters*—materials not requiring evaporation, that are used to remove gases present after device exhaust. The gases may be generated during vacuum device processing or operation, or both.

3.1.2 *surface getter*—a getter where the surface is strictly dominant and the getting rate and sorption capacity per unit area are essentially independent of the thickness at operating pressure and temperature.

3.1.3 *volume getter*—a getter where the getting rate or sorption capacity per unit mass, or both is dependent on the thickness at operating pressure and temperature.

3.1.4 *activation*—the conditioning by thermal treatment of a getter to develop its getting characteristics.

3.1.5 *reactivation*—any conditioning by thermal treatment of the getter subsequent to activation which at least partially restores its getting characteristics.

3.2 *gas content*, GC, of a getter can be classified as:

3.2.1 *total gas content*, TGC—*of a getter*, the sum total of the gases in or on the getter, chemically or physically bound or in solution.

3.2.2 *total hydrogen content*, THC—*of a getter*, the total quantity of hydrogen in solution.

3.2.3 *hydrogen gas content*, HGC—the quantity of hydrogen evolved when a getter is heated from room temperature to its activation temperature.

3.3 *reactivation gas content*—the quantity of gas evolved from a getter on reactivation.

<sup>4</sup> Available from the American Vacuum Society, 120 Wall St., 32nd Fl., New York, NY 10005.



3.4 *Sorption* by a getter is the process of removing gases from a vacuum device by adsorption or absorption phenomena.<sup>5</sup>

3.4.1 *Adsorption* describes gas interactions at the surface of the getter material. These may be either physical or chemical.

3.4.2 *Absorption* deals with gas interactions within the bulk of the getter material and is dependent on porosity, diffusion rate, solubility, chemical reactions, temperature, and pressure.

3.4.3 Certain gases may act reversibly with getter materials. Examples of this are the reaction of hydrogen with titanium or zirconium. These gases may be released upon reactivation and removed by pumping if desired.

3.4.4 Quantities for released or sorbed gases are measured in torr litres (pascal cubic metres) at  $23 \pm 2^\circ\text{C}$ .

3.5 *getter pumping speed, G*—the volume of gas sorbed per unit time. It is measured in litres per second (cubic metres per second).

3.6 *initial getter pumping speed, G<sub>i</sub>*—the instantaneous gettering rate 3 min after the start of the test at the chosen pressure and temperature. The time delay is necessary to allow initial transient effects to become negligible. This time delay may be modified as required and should be reported.

3.7 *terminal getter pumping speed, G<sub>T</sub>*—the rate at which the getter pumping speed decreases to 5 % of the initial getter pumping speed unless otherwise specified.

3.8 *gas sorption capacity, C*—the quantity of gas sorbed by the getter while it is at operating temperature until the terminal getter pumping speed is reached. This quantity is expressed in torr litres (pascal cubic metres). The gas sorption capacity is rarely coincident with the stoichiometric capabilities under operation conditions. Consequently, reactivations are usually possible.

3.9 *residual gettering characteristics*—the sorption capacity and getter pumping speed for another gas after the terminal gettering rate has been reached for a previous gas specie. Displacement of the prior test gas specie may occur and should be considered.

3.10 *reserve gettering characteristics*—the sorption capacity and getter pumping speed for a given gas after the initial terminal getter pumping speed has been reached and the getter reactivated.

3.11 *mass throughput, Q*—the quantity of gas flowing through a given plane in unit time at a given temperature. It is measured in torr litres per second (pascal cubic metres per second).

3.12 *molecular flow region*<sup>6</sup>—that pressure region where gases or vapors flow under conditions such that the largest internal dimensions of a transverse section of the vessel is many times smaller than the mean free molecular path. Under these conditions the rate of flow is limited by collisions of molecules with walls and not by collisions between molecules. The molecular flux is not necessarily isotropic in molecular flow.

3.13 *conductance, F*—of a system for a given gas—the ratio of throughput  $Q$  for a given gas to the pressure difference across the system,  $(P_2 - P_1)$ , in the steady state. It is measured in litres per second (cubic metres per second) and in the molecular flow region is given by  $F = Q/(P_2 - P_1)$  where  $P_2$  is the upstream pressure and  $P_1$  is the downstream pressure.

3.14 *Getter Materials:*

3.15 *active getter material*—an element, alloy, compound, or mixture thereof, on and within which significant gettering occurs.

3.16 *impurities-in getters*—the weight percents of elements or compounds that may or may not significantly affect getter characteristics.

3.17 *contamination*—the process whereby the getter adversely affects what is around it, that is, the device or system.

3.18 *poisoning*—the process whereby the environment around the getter, that is the system or device, adversely affects the getter.

3.19 *getter mount*—a mechanical device used to secure the getter and its integral support leg(s), if any, at the specified position in the getter test bulb.

3.20 *getter test chamber*—that portion of the apparatus in which the getter is mounted and tested.

3.21 *gettering rate*—the mass of gas absorbed per unit of time.

#### 4. Hazards<sup>7</sup>

4.1 These practices should be accomplished only by properly trained and qualified personnel as there may be problems in toxicity, combustion, implosion, explosion, and in some cases radioactivity. Safety precautions should be observed in the use of corrosive, toxic, and flammable gases and in the design and operation of the vacuum test apparatus.

4.2 *Recommended Getter Handling Precautions*—Possible toxic problems associated with ingestion, inhalation, skin contact, or radioactivity should be investigated. Generally a finished getter product is relatively safe and easily handled since most nonevaporable getters are metallic powders in a sintered or otherwise bonded form. The major concern results from the large surface area to volume ratio, which makes it possible to ignite the material in air at some temperature that is determined by the particular composition of the getter.

4.3 *Commercially Purchased Getters*—In all cases manufacturer's literature should be a guide for safe handling. Care should be exercised in storage, cleaning, and processing of the getter. The finished product can be ignited and could combine chemically with certain acid, alkaline, or organic materials resulting in possible dangerous reactions.

4.4 *Experimental Production or Manufacture of Getters*—Since nonevaporable getters are generally made from metal powders, only those persons trained in safe handling of fine reactive powders should be involved with their fabrication. The obvious hazards of metal powder explosion, fire, and the potential detrimental effects of eye and lung contact make extreme caution imperative.

<sup>5</sup> Redhead, Hobson, and Kornelsen, *The Physical Basis of Ultrahigh Vacuum*, First Edition, Chapman and Hall, Ltd, London, England.

<sup>6</sup> Dushman and Lafferty, *Scientific Foundation of Vacuum Techniques*, Second Edition, John Wiley & Sons, Inc., New York, NY.

<sup>7</sup> Sax, H. L., *Dangerous Properties of Industrial Materials*, Fourth Edition, Van Nostrand Reinhold Co., New York, NY.



## 5. Test Specimen—Activation and Characterization

5.1 Test specimens are usually commercial nonevaporable getters. The major components are the active material, the substrate or container, and its support, or combination thereof.

5.1.1 *Nonevaporable getters* come in a variety of forms. The active bulk getter materials may be in the form of bars, chips, powders, sheets, strips, washers, or wire. These materials may be employed to fill suitable containers, compacted into pressed pellets, sintered into or on supporting bodies, or used for form coatings on a suitable substrate.

5.1.2 *Active Metal Characterization*—A nonevaporable getter is characterized by its gettering rate, sorption capacity, optimum operating temperature, and activation parameters (time – temperature) and the gases sorbed. The gases specified as standard test gases are hydrogen and carbon monoxide. These gases are representative of gases that reversibly and irreversibly react with the getter material but do not represent sorption characteristics for other gases or gas mixtures.

### 5.2 *Getter Handling:*

5.2.1 Getters should be handled only with clean tools, rubber or plastic gloves or finger cots, never with bare hands or woven gloves.

5.2.2 *Storage*—For long-term storage a clean, dry ambient is desirable. Getters may be stored in a phosphorus pentoxide or a silica gel air desiccator or under a dry inert gas atmosphere.

### 5.3 *Getter Activation:*

5.3.1 The activation parameters are temperature, pressure, time, and method of heating. Maximum allowable temperature, pressure, and time that will not degrade getter sorption characteristics should be provided by the manufacturer. Activation should be initiated under high vacuum conditions of approximately  $1 \times 10^{-6}$  torr ( $1 \times 10^{-4}$  Pa) or lower pressure to protect gettering characteristics. The heating rate of the getter should be controlled to avoid excessively high system pressure due to outgassing. Care should be exercised to avoid premature partial activation of the getter material when high gas load conditions exist. Activation can be classified as one of two types. The first type should be used to determine the inherent gettering characteristics of a given material. This activation should be accomplished under conditions judged most nearly optimum by the manufacturer. The second type of activation is one that is not aimed at determining inherent material characteristics, but rather is dictated by application limitations. It is accomplished under conditions different from the recommended parameters. In all cases, in order to correlate test data, careful attention must be given to reproducing all bake and activation conditions. Nonevaporable getters are activated by heating the getter to its activation temperature for the activation time. The activation temperature and time are functions of the getter material and the activation technique.

5.3.2 Activation may be accomplished by induction heating, joule (resistance) heating, radiant heating, conductance heating, electron bombardment, etc. The temperature must be monitored while the activation process is in progress. Thermocouples, properly selected and used, are the preferred temperature sensors.

### 5.4 *Getter Identification:*

5.4.1 The getter part number using the getter manufacturer's nomenclature identifies the particular getter used.

5.4.2 The getter lot number identifies the manufacturer's production batch and production date. From the lot number and the manufacturer's control charts, it shall be possible to trace all production cycles to incoming raw materials.

## 6. Dynamic Gas Sorption Characteristics of a Nonevaporable Getter

6.1 The sorption efficiency of a getter device is determined by the gettering and sorption capacity. These are determined dynamically from the instantaneous values of gas throughput into the getter after the getter has been activated and is operating within the test temperature range. The test gas being gettered is made to flow through the known conductance. The gettering and the instantaneous gas throughput can be calculated knowing the conductance and the pressure drop across it. Integrating the instantaneous throughput over the time of the test gives the quantity sorbed. The standard test gases are carbon monoxide and hydrogen as representative of irreversibly and reversibly gettered gases. Additional data on other getterable gases may be supplied by the manufacturer on request. For specific applications other test gases may be mutually agreed upon between the seller and the user. There are three broad areas of application of gas sorption measurements: basic studies of gettering properties, getter performance in a specific vacuum device, and comparison between getter types. When basic studies of sorption mechanisms or calculation of activation energies are required, the test should be performed with a constant pressure above the getter since the diffusion of gas into the interior of the getter is the rate limiting factor, and diffusion depends on pressure and temperature. This practice recommends the use of constant pressure above the getter in all cases. It should be noted that getter evaluation tests may be carried out with either constant manifold pressure or constant throughput; however, the results may not in general be comparable.

### 6.2 *Problems and Pitfalls:*

6.2.1 When making measurements of sorption characteristics, strict adherence to the following paragraphs should be observed.

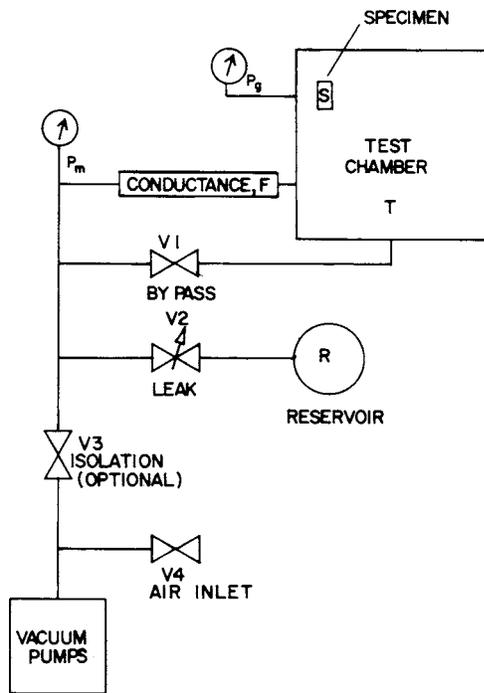
6.2.2 The sorption characteristics are adversely affected by foreign gases present as impurities in the test gas or emitted by the apparatus, or both. For conventional test procedures, the test gas should be at least 99.99 % pure. The apparatus must be capable of reaching an ultimate pressure of less than  $1 \times 10^{-8}$  torr ( $1 \times 10^{-6}$  Pa). In practice, this requires a bakeable system. Vacuum pumps having minimal selective action, such as adequately trapped diffusion pumps or turbomolecular pumps, are preferred. However, the more selective pumps may be used provided that their pumping speeds for both system and test gases are much greater than known system conductances. Residual gas analysis (see AVS Recommended Practice 2.3) should be used in this case. The getter and the pressure gage should be located so as to minimize wall heating.

6.2.3 The pressure gages (see Practice E 296 and Test Method E 297) for both the getter and manifold pressures should be of the Bayert-Alpert type and used with a maximum electron ionizing current of 10  $\mu$ A. A low-temperature electron

emitter is preferred. Larger ionizing currents can be used provided that there is no evidence of pumping of the gases after saturation (see 6.5.4) and before start of test. Gage pumping should be less than 5 % of the terminal gettering rate of interest. The gages must be calibrated against an absolute gage or an adequate transfer standard. See AVS Recommended Practices 6.2, 6.4, and 6.5. Excessive degassing of the electron collector may result in unwanted pumping as well as a shift in gage calibration. A calibrated residual gas analyzer can be used to measure the pressure above the getter.

6.2.4 The known conductance,  $F$ , is chosen in relation to the type of getter (in particular anticipated gettering rate) and the measuring system. The conductance value must ensure that the manifold pressure is sufficiently low, initially, that molecular gas flow through the conductance applies. The conductance value must be sufficiently low to allow measurement of the 5 % terminal gettering rate. The conductance,  $F$ , must be such that the pressure above the getter,  $P_g$ , is accurately measurable and therefore considerably greater than the residual system pressure (see Annexes).

6.3 The dynamic gas sorption apparatus is shown schematically in Fig. 1. During a test the bypass valve,  $V1$ , is closed. The leak valve,  $V2$ , maintains a constant pressure,  $P_g$ , above the getter. Part of the inflowing test gas is continuously pumped by the vacuum pumps to minimize buildup of slowly pumped gases while the remainder flows through the known conductance  $F$  and is removed by the getter. Valve  $V3$  may be used as



NOTE 1—Incoming gases must: (1) impinge on walls before reaching getter and (2) flow over getter before reaching pressure sensor  $P_g$ .  
NOTE 2—The conductance between the getter and the test chamber must be as large as possible.

FIG. 1 Dynamic Gas Sorption Apparatus

an isolation valve in determining real and virtual leak rates. Fig. 2 and Fig. 3 show examples of systems used for these measurements.

6.4 Selection of Working Parameters—Several factors must be considered in selecting the parameters for testing a specific getter, as follows:

6.4.1 The initial getter pumping speed is obtained from the manufacturer’s data and is referenced to specific operating temperatures.

6.4.2 The pressure above the getter,  $P_g$ , is obtained by making a reasonable estimate of the expected initial getter pumping speed,  $G_i$ , and the total getter capacity,  $C$ . In the equation:

$$C = \int_0^t P_g G(t) dt, \quad (1)$$

If  $G(t)$  is known, Eq 1 can be integrated to obtain the area under the  $Q$ -versus- $t$  curve. For sizing purposes, assume  $G$  decreases linearly from  $G_i$  to 0 in time  $t$ . Integrating and rearranging Eq 1,

$$P_g t = 2C/G_i \quad (2)$$

The higher the value of  $P_g$ , the shorter will be the test time. For  $C$  in torr litres,  $G_i$  in litres per second, and a 3-h test time,

$$P_g = 2Ct/G_i = C/5400G_i \quad (3)$$

It is desirable that  $P_g$  be as close to anticipated use pressure as practical, within the limits of total test time, since the diffusion of the test gas into the bulk of the getter can be the rate limiting factor. In the absence of guidelines, it is suggested that  $P_g$  chose between  $1 \times 10^{-5}$  and  $1 \times 10^{-6}$  torr ( $1 \times 10^{-3}$  and  $1 \times 10^{-4}$  Pa) and preferably at  $3 \times 10^{-6}$  torr ( $4 \times 10^{-4}$  Pa).

6.4.3 The initial ratio of manifold pressure to the pressure above the getter,  $P_m/P_g$ , must be at least 40 so that the difference between  $P_m$  and  $P_g$  is readable when the terminal gettering rate (5 % of  $G_i$ ) is reached. The maximum manifold pressure must be within the range of the pressure sensor used and molecular flow must hold.

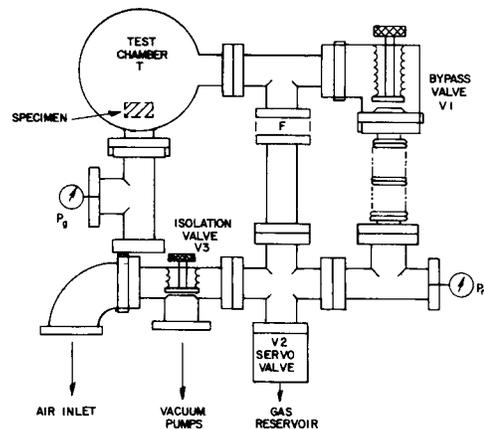


FIG. 2 Dynamic Gas Sorption Apparatus for Large Surface Area Getters