Designation: F 798 – 97

Standard Practice for Determining Gettering Rate, Sorption Capacity, and Gas Content of Nonevaporable Getters in the Molecular Flow Region [1]

1. Scope

1.1 This practice describes techniques for determining gettering 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 ±15 % for gettering rate and sorption capacity. Multilaboratory reproducibility is estimated at ±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 [2]
E 297 Test Method for Calibrating Ionization Vacuum Gage Tubes [3]
2.2 *American Vacuum Society Standards:*

Recommended Practice 2.3 Procedure for Calibrating Gas Analyzers of the Mass Spectrometer Type [4]
Recommended Practices 6.2, 6.4, and 6.5 Procedures for Calibrating Pressure Gages and Their Controls [4]

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 gettering 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 gettering 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 gettering characteristics.

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

3.4 *Sorption* by a getter is the process of removing gases from a vacuum device by adsorption or absorption phenomena. [5]

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 $23 \pm 2°C$.

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_i*—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_T*—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*[6]—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 throughout 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 *3.22. gettering rate*——the *mass* of gas absorbed per unit of time.

4. Hazards [7]

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

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[5] Redhead, Hobson, and Kornelsen, *The Physical Basis of Ultrahigh Vacuum*, First Edition, Chapman and Hall, Ltd, London, England.

[6] Dushman and Lafferty, *Scientific Foundation of Vacuum Techniques*, Second Edition, John Wiley & Sons, Inc., New York, NY.

[7] Sax, H. L., *Dangerous Properties of Industrial Materials*, Fourth Edition, Van Nostrand Reinhold Co., New York, NY.

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.

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 1×10^{-6} torr (1×10^{-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 1×10^{-8} torr (1×10^{-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 µA. 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 measureable 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
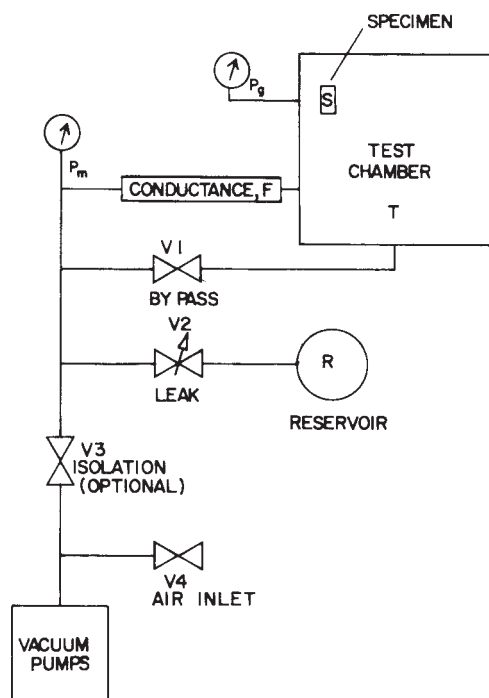
the getter. Part of the inflowing test gas is continuously pumped by the vacuum pumps to minimize buildup of slowly pumped gases while the remainder flows through the known conductance F and is removed by the getter. Valve $V3$ may be used as an isolation valve in determining real and virtual leak rates. 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:

$$C = \int_t P_g G(t)dt, \tag{1}$$

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,

$$P_g t = 2C/G_i \tag{2}$$

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,

$$P_g = 2C/tG_i = C/5400G_i \tag{3}$$

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 1×10^{-5} and 1×10^{-6} torr (1×10^{-3} and 1×10^{-4} Pa) and preferably at 3×10^{-6} torr (4×10^{-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

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.

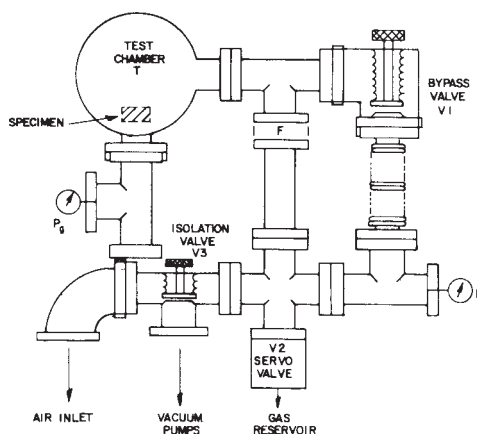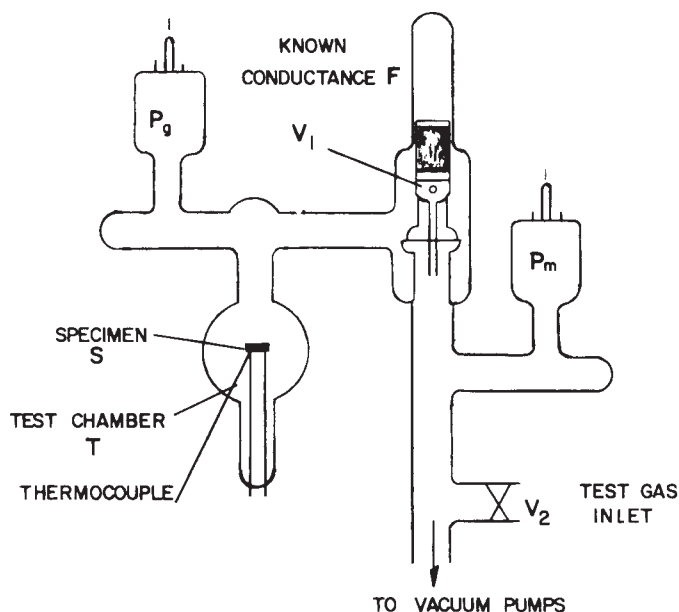**FIG. 1 Dynamic Gas Sorption Apparatus**

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

NOTE 1—Known conductance and *VI* is composed of a grease-free spherical ground-glass joint actuated magnetically. Which open it is the "by-pass." When closed it is the "conductance."

FIG. 3 All Glass Dynamic Gas Sorption Apparatus for Surface and Volume Getters

is a prime factor in selecting the conductance. The problem should not be over-emphasized since it is at the end of the test where the major results are already determined. However, some ratio, R, must be chosen and a number between 2 and 10 is reasonable. Substituting $R\,P_g = P_m$ in

$$(P_m - P_g)\,F = G\,P_g \quad \text{and at} \quad G = 0.05G_i \tag{4}$$

$$(R - 1)\,P_g\,F = 0.05G_i\,P_g \tag{5}$$

$$F = 0.05G_i\,/\,(R - 1) \tag{6}$$

Eq 6 allows one to adjust the conductance to a convenient value by varying R between 2 and 10.

6.4.5 Fig. 4, a plot of P_m/P_g versus G with conductance as a parameter, facilitates selection of conductance values. For example for a P_m/P_g ratio of 40 and a G_i of 10 L/s, a conductance of 0.25 L/s is indicated. For P_g of 3×10^{-6} torr, the initial P_m is 1.2×10^{-4} torr and the terminal P_m, 9×10^{-6} torr.

6.4.6 The maximum manifold pressure, P_{mi}, occurs at the start of the test and can be estimated by:

$$(P_{mi} - P_g)\,F = G_i\,P_g \tag{7}$$

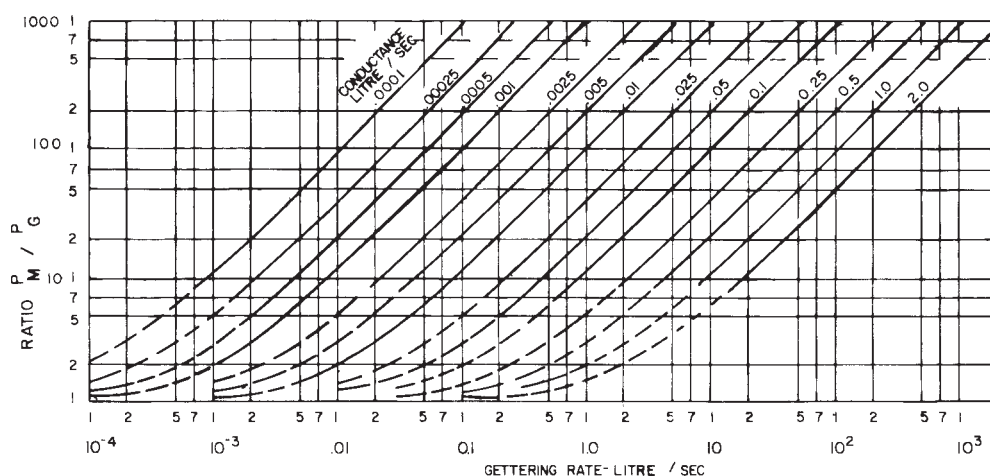Substituting $F = 0.05G_i/(R - 1)$ from Eq 6 and $P_g = 2C/tG_i$ from Eq 3 and rearranging:

$$P_{mi} \approx 40\,(R - 1)\,C/tG_i \tag{8}$$

For $t = 3$ h, $P_{mi} \approx (R - 1)\,C/270G_i$ torr.

6.5 *Procedure*:

6.5.1 Mount the getter in the dynamic gas sorption apparatus, observing clean handling procedures.

6.5.2 Bake while pumping to ensure that a pressure of less than 1×10^{-8} torr (1×10^{-6} Pa) is reached in the getter chamber after cooling to room temperature. Significant partial activation of the getter material must be avoided. The getter temperature should be monitored during the bake.

pressure must be within the range of the pressure sensor used and molecular flow must hold.

6.4.4 The test conductance F must be smaller than the system conductances: a ratio of system conductance to test conductance of at least 100 is desirable. As the terminal getter pumping speed is approached, the pressure P_m achieves its lowest value and approaches P_g, and the measurement of the difference $(P_m - P_g)$ across the conductance becomes subject to gaging error problems. Thus the minimum ratio of P_m to P_g

NOTE 1—Derived from

$$Q = F\,(P_M - P_G) = GP_G$$
$$P_M/P_G = G/F + 1$$

NOTE 2—For a given initial getter pumping speed and a desired pressure above the getter, select $AP_M \cdot P_G$ ratio. This choice is limited by two factors: the max P_M must be within the range of the pressure sensor and at the terminal getter rate (5 % of initial) $P_M \geq 2P_G$ for readability.

FIG. 4 Ratio of Manifold Pressure to Pressure Above the Getter (P_M/P_G) verses Getter Pumping Speed, G, for Various Conductance, F

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6.5.3 It is reasonable to require that the system gas evolution be small compared to the mass flow into the getter. The system gas evolution rate may be measured by isolating the system from the vacuum pumps using valve *V3* if present and measuring the pressure rise, after the initial burst, on the P_g ionization gage. The worst point is at the final portion of the test where $G = 0.05G_i$.

$$G_F P_g >> V_T\, dP/dt \qquad (9)$$

where V_T is the chamber volume and dP/dt is the rate of pressure rise when the test chamber is blanked off. If a 1:100 ratio is required between gassing and gettering,

$$0.05dG_i P_g \geq 100V_T\,(dP/dt) \qquad (10)$$

Since

$$P_g = 2C/tG_i$$

then

$$0.1C/t \geq 100V_T\,(dP/dt) \qquad (11)$$

or

$$dP/dt\, C/1000t\, V_T \qquad (12)$$

which points out the desirability of high P_g testing or relatively short tests. Again for a 3-h test $dP/dt \leq (C/1.08V_T)10^{-7}$ torr/s. If a sufficiently low gas evolution rate (combined real and virtual leaks) has been assured, open the system to the vacuum pumps and degas the pressure gages.

6.5.4 At the beginning of the test, compare the gages with respect to each other by allowing the test gas into the system. This should saturate the gages and system walls with the test gas and hence minimize gage and wall pumping effects. Under no circumstances should further gage degassing be carried out. Each decade within the measurement range must contain at least three check points for the test gas. The comparison calibration should start at the highest pressure and proceed to the lowest. Due to limitations of the currently available gages such as the McLeod or capacitive manometers, calibration on this apparatus is not possible. Spinning rotor gages should be used.

6.5.5 *Getter Activation*:

6.5.5.1 Pump until a pressure P_g of less than 1×10^{-6} torr (1×10^{-4} Pa) is reached.

6.5.5.2 Activate the getter by heating to activation temperature following manufacturer's recommendations for temperature and time.

6.5.5.3 Monitor the maximum pressure during activation using only the P_g gage. This gives a relative value of the gas content of the getter.

6.5.5.4 Allow the getter to cool to its test temperature. Then close valve *V1*. Using the selected conductance admit the test gas so that the required P_g is established in the shortest time possible, less than 60 s. Immediately record P_m. Maintain P_g constant, using valve *V2* and begin recording P_m. Terminate the test when the gettering rate has reached the calculated terminal value based upon the initial measured gettering rate. The anticipated P_m when the terminal gettering rate is reached can be obtained from Fig. 4 or is calculated knowing the conductance and the initial gettering rate.

6.6 The equations used to calculate gettering rate, instantaneous throughput, and quantity sorbed are given below:

$$\begin{aligned}Q_i &= F\,(P_m - P_g)\\ &= G\,P_g \quad \text{torr litre per second}\end{aligned}$$
$$\text{(pascal cubic metre per second)} \qquad (13)$$

Therefore

$$G = F\,(P_m - P_g)/P_g \text{ litres per second}$$
$$\text{(cubic metres per second)} \qquad (14)$$

where:
Q_i = instantaneous throughput, torr·L/s (Pa·m^3/s),
F = known conductance, L/s (m^3/s),
P_m = manifold pressure, torr (Pa),
P_g = pressure above getter, torr (Pa), and
G = getter pumping speed, L/s (m^3/s).

and

$$C\int_t = Q_i\, dt \qquad (15)$$

where:
C = quantity sorbed in time t, torr·L (Pa·m^3).

6.7 *Data Presentation*:

6.7.1 The getter pumping speed and sorption capacity for a given getter system are calculated during the equations of 6.6. Since the getter pumping speed is a function of the quantity of gas sorbed, the gettering characteristics of a given getter can be displayed graphically using a log-log plot. Display the log getter pumping speed G as ordinate versus the log quantity of gas sorbed C as abscissa. Fig. 5 and Fig. 6 presents plots for various types of getters.

6.7.2 Fig. 7, Suggested Report Sheet, lists pertinent information that should be reported. Note that gettering rates, sorption capacity, and gas content are reported for the given getter and may be normalized to unit area or unit mass depending on the getter type.

7. Gas Content of a Getter

7.1 *Significance*—It is normally complex to determine total gas evolution from all classes of nonevaporable getters in a quantitative and reproducible manner. The evolution of hydrogen from certain nonevaporable getter materials (such as zirconium, titanium, thorium, etc., and their alloys) is easily determined. However other gases and contaminants (such as chlorine, fluorine, sulfur, etc.) can be determined only by using more sophisticated techniques. The total hydrogen gas content of the getter cannot be accurately measured. However, the hydrogen gas, HGC, released during activation is proportional to the total hydrogen content and is useful in designing the exhaust system and defining getter activation schedules. The hydrogen gas content, HGC, may also be useful in quality assurance.

7.2 *Summary of Method*—The hydrogen gas content, HGC, is determined by slowly heating the getter from room temperature to its activation temperature in a closed system of known volume at room temperature, and measuring the quantity of gas evolved. The gas may be collected in several steps if necessary.
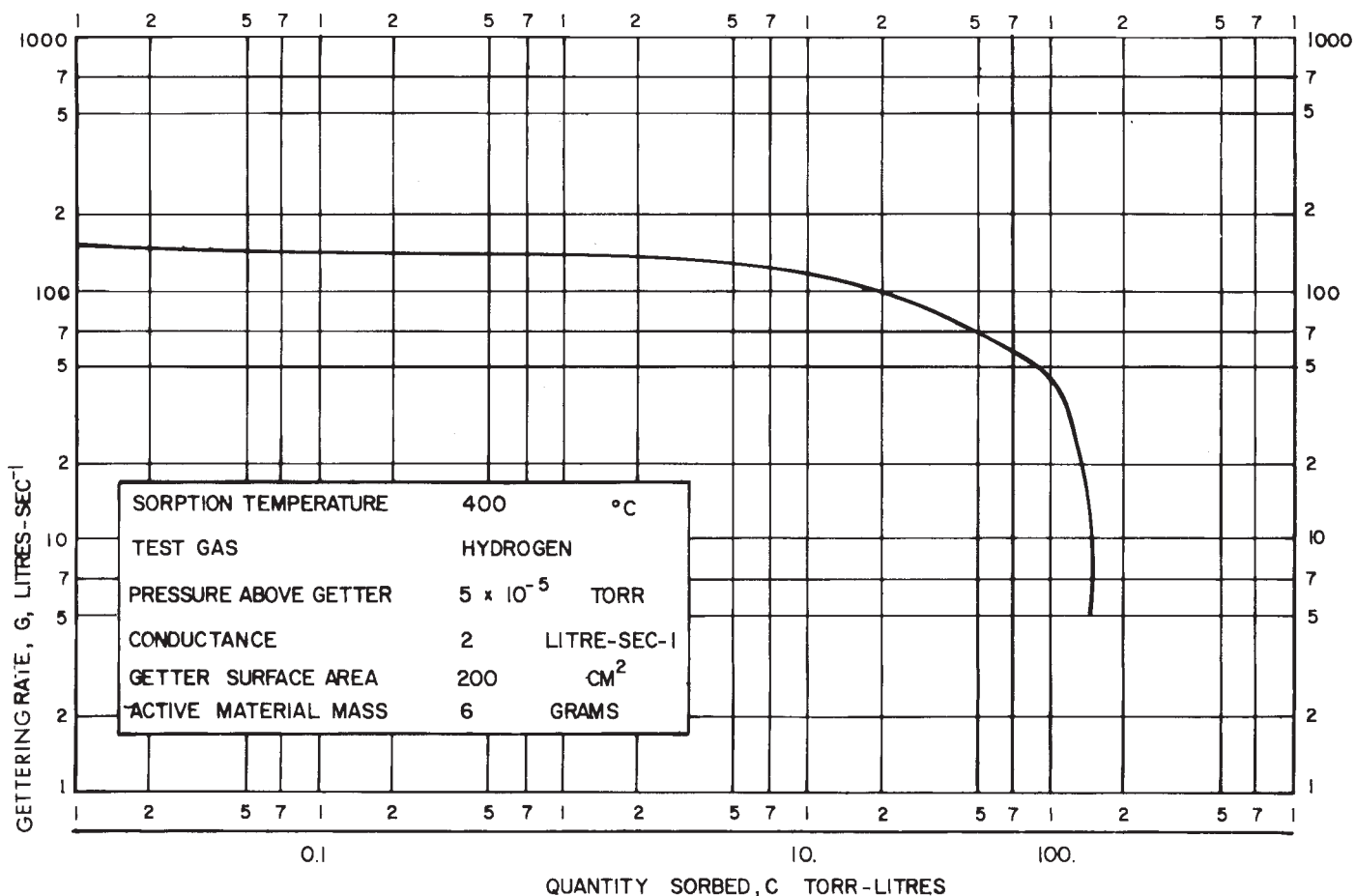
7.3 *Problems and Pitfalls*:

FIG. 5 Getter Pumping Speed, *G*, verses Quantity Sorbed, *C*, for a Large Surface Area Getter

7.3.1 The getter device must be held at its activation temperature and not be allowed to cool during the measurement since, in general, the getter will sorb some, if not all, of its evolved gases on cooling.

7.3.2 The liquid nitrogen trap should be kept filled during the test since it acts as a selective pump for the condensible gases.

7.3.3 The test chamber diameter is chosen as two to three times the sample diameter as a compromise between radiant heating of the wall and minimizing the test chamber volume.

7.3.4 When induction heating is employed, plasma discharges must be avoided.

7.3.5 Frequent pressure gage calibration is mandatory.

7.3.6 Getter temperatures must be measured to ±5°C. A thermocouple is the preferred temperature sensor. The thermocouple used must not alloy with the getter used and should have minimal heat conductance. Chromel-Alumel thermocouples with 0.2 to 0.3-mm diameters have been successfully used.

7.4 *Apparatus*—A typical hydrogen gas content test apparatus is shown in Fig. 8. It consists of a known volume in which the getter is mounted and activated and which is capable of being isolated from the pumping system. Available experience and the tests described below have been made on glass systems. There are three pressure gages: a conventional ionization gage, *G1*, and two gas content gages, *G2* and *G3*, all of which should have sufficiently fast response times and possess stable calibrations with time and repeated exposure to air. The following gas content gages are recommended: Pirani, capacitive manometer, or the McLeod gage.

7.4.1 The ionization gage, *G1*, is used at the lower pressures. The gas content pressure gages, *G2* and *G3*, must read accurately in the 0.01 to 0.1-torr range. The gas content gages should preferably be independent of gas specie. However, if a specie-dependent gage is used, it should be calibrated for hydrogen.

7.4.2 The total volume of the system and the volume ratio of the test chamber and the reservoir are chosen such that the pressure, P_{fn}, is in the range of greatest accuracy for the instrumentation used. This volume must be determined to 5 % or better.

7.5 *Procedure*:

7.5.1 Mount the getter in the gas content apparatus, observing clean handling procedures.

7.5.2 Evacuate the system to less than 1×10^{-5} torr (1×10^{-3} Pa).

7.5.3 Then fill the glass trap with liquid nitrogen.

7.5.4 Upon closing valve *V3 (V2* open and *V1* closed), to isolate the system from the vacuum pumps, the pressure should
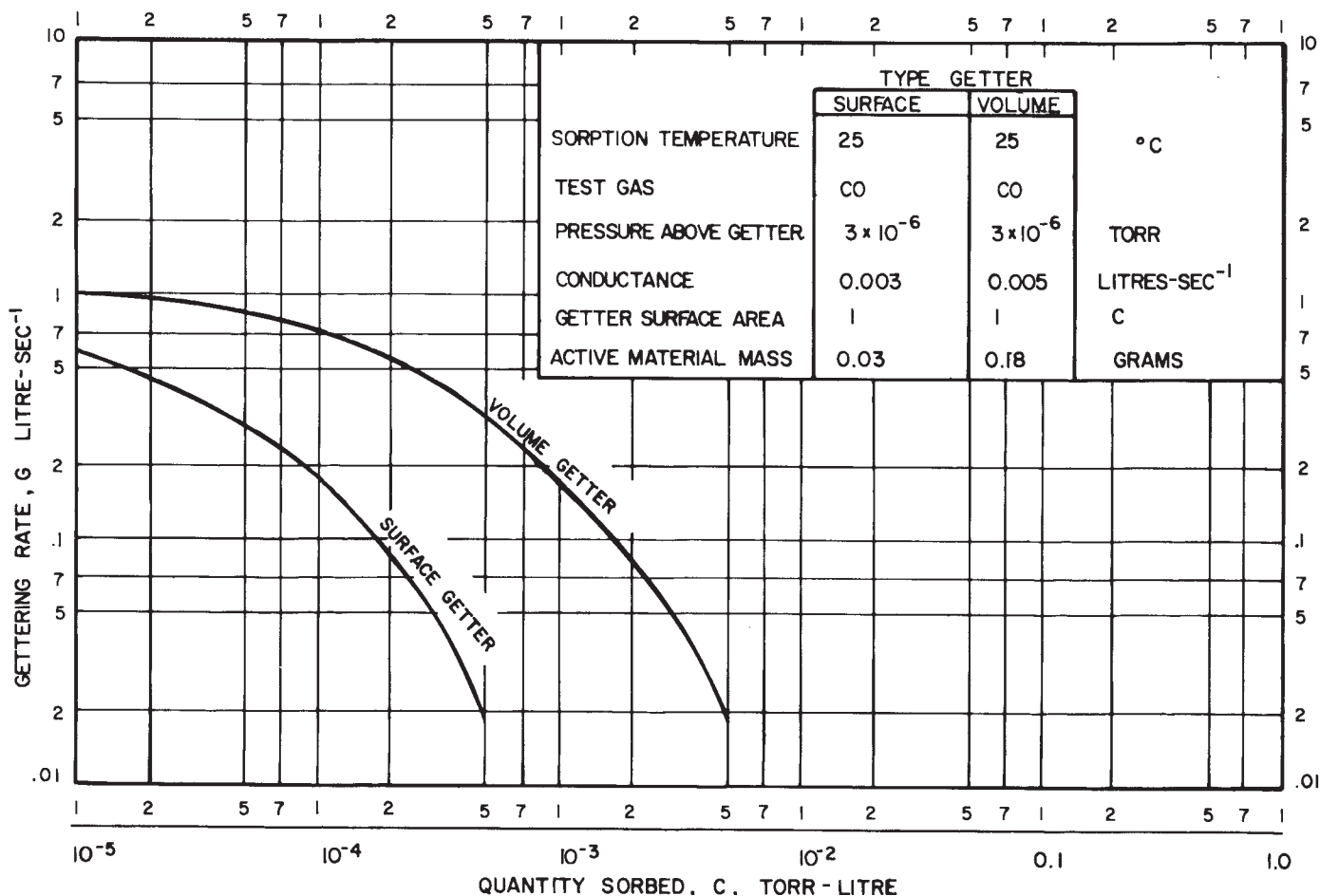
ASTM F 798 – 97

FIG. 6 Getter Pumping Speed, *G*, verses Quantity Sorbed for a Surface Area and a Volume Getter

GETTER TYPE
  Manufacturer's Nomenclature_____
  Manufacturer's Lot Number_____
  Production Date_____
  Purchase Order No._____ Date_____
  User Batch Number _____
  Geometric Surface Area of Material _____ cm^2(m^2)
  Mass of active material _____ g (kg)
GETTER TEST PARAMETERS
  Conductance L/s_____ (m^3/s)
  Test Gas   Type _____ Purity_____ %
  Pressure above Getter _____ torr (Pa)
  Operating Temperature _____° C
  Activation Temperature _____° C
TEST RESULTS
  Initial Getter Pumping Speed L/s _____ (m^3/s)
  Terminal Getter Pumping Speed _____ L/s (m^3/s)
  Sorption Capacity _____ torr·L Pa·m^3
  Hydrogen Gas Content _____ torr·L (Pa·m^3)
NORMALIZING TEST RESULTS
  The test results on individual getters may be normalized and reported per unit
area for surface getters and per unit mass for volume getters.
NOTES
  Any deviation from manufacturer's recommended activation and operating
conditions or deviations, or both, from this test procedure should be included as
part of this report.

FIG. 7 Suggested Report Sheet

not go above 5.0×10^{-4} torr (6.5×10^{-2} Pa) during a period of time at least equal to that required to perform the test.

7.5.5 Heat the getter slowly to its activation temperature with all valves closed. When the pressure in the test chamber

8

F 798 – 97
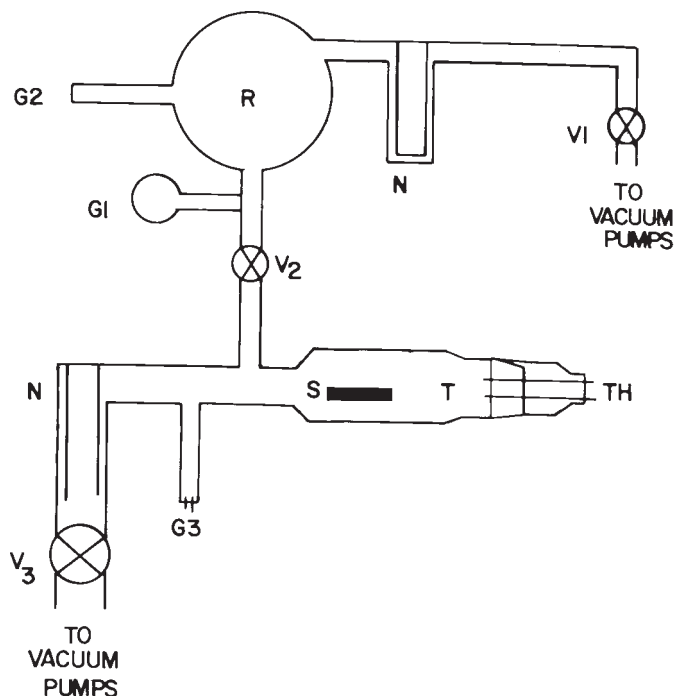
G1	ionization gage
G2	gas content gage
G3	gas content gage
N	liquid nitrogen trap
R	reservoir
S	specimen
T	test chamber
TH	thermocouple
V1	valve
V2	valve
V3	valve

FIG. 8 Typical All-Glass Hydrogen Content Apparatus

reaches a value no longer compatible with accurate measurement on *G3*, open valve *V2* and expand the gas into the reservoir *R*. Close *V2* and continue to collect gas in the test chamber. Determine the pressure in the reservoir using *G2* and then evacuate *R* by opening *V1*. The collection of gas is continued until such time as the quantity of gas collected in *R* is less than 5 % of the gas already collected.

7.5.6 Remove the liquid nitrogen from the traps and warm before admitting air to the system.

7.6 *Calculations*—Calculate the hydrogen gas content, HGC, as follows:

$$HGC = V_R\sum_1^n (P_{fn} - P_{in}) - H + V_T (P_{ff} - P_{io})$$

where:
V_R = reservoir volume, L (m^3),
P_{fn} = observed reservoir pressure when filled in nth filling, torr (Pa),

P_{in} = observed reservoir pressure when evacuated in nth filling, torr (Pa),
H = uncompensated for volume of gas leaked into system during test,
V_T = test chamber volume, L (m^3),
P_{ff} = final test chamber pressure, torr (Pa), and
P_{io} = initial test chamber pressure, torr (Pa).

or:

$$HGC \times V_R \sum_{n=1}^n P_{fn} \quad \text{torr·L (Pa·m}^3)$$

when:

$$P_{in} << P_{fn},$$

H conforms to requirements of 7.5.4 and $V_t(P_{ff} - P_{io}) \times$ HGC.

8. Keywords

8.1 getter pumping speed; hydrogen content measurements; non-evaporable getters;

9

F 798 – 97

ANNEXES

(Mandatory Information)

A1. CALCULATION OF MOLECULAR FLOW CONDUCTANCE

A1.1 Aperture

$$F_0 = \alpha A \, (T/M)^{1/2}$$

where:
F_0 = conductance, L/s (m^3/s),
A = area, cm^2 (m^2),
T = temperature, K,
M = molecular weight, g/mol (kg/mol), and
α = 3.638 if the Cgs units indicated are used, or
α = 1.150 if SI units indicated in parentheses are used.

A1.2 Long Uniform Cylindrical Tube 1/a > 100

$$F = \beta a^3/l \, (T/M)^{1/2}$$

where:

F = conductance, L/s (m^3/s),
a = radius, cm (m),
l = length, cm (m), and
β = 30.48 if the Cgs units indicated are used or
β = 9.64 if the SI units indicated in parentheses are used.

A1.3 Short Uniform Cylindrical Tube

$$F_t = \alpha \pi a^2 K \, (T/M)^{1/2}$$

where:
F_t = conductance, L/s (m^3/s),
K = Clausing's correction factor given in Table A1.1, and
α = 3.638 if the Cgs units indicated are used or
α = 1.150 if SI units indicated in parentheses are used.

10

TABLE A1.1 K values for *l/a*

l/a	K	*l/a*	K
0	1	3.2	0.4062
0.1	0.9524	3.4	0.3931
0.2	0.9092	3.6	0.3809
0.3	0.8699	3.8	0.3695
0.4	0.8341	4.0	0.3589
0.5	0.8013	5	0.3146
0.6	0.7711	6	0.2807
0.7	0.7434	7	0.2537
0.8	0.7177	8	0.2316
0.9	0.6940	9	0.2131
1.0	0.6720	10	0.1973
1.1	0.6514	12	0.1719
1.2	0.6320	14	0.1523
1.3	0.6139	16	0.1367
1.4	0.5970	18	0.1240
1.5	0.5810	20	0.1135
1.6	0.5659	30	0.0797
1.7	0.5518	40	0.0613
1.8	0.5384	50	0.0499
1.9	0.5256	60	0.0420
2.0	0.5136	70	0.0363
2.2	0.4914	80	0.0319
2.4	0.4711	90	0.0285
2.6	0.4527	100	0.0258
2.8	0.4359	1000	0.002658
3.0	0.4205	...	$8a/3l$

A2. MEASURING THE CONDUCTANCE OF THE APPARATUS

A2.1   Using the dynamic gas sorption apparatus shown schematically in Fig. 1. Without a getter specimen, hold the manifold pressure, P_m, constant and measure the pressure rise in the test chamber of volume V_T.
Then:

$$Q = V_T \, (dP/dt) = (P_m - P_g) \, F$$

and

$$\int_i^f (dP_g)/(P_m - P_g) = F/V_T \int_i^f (dt), \, t_i = 0$$

Integrating,

$$F = (V_T/t) \ln \left[(P_m - P_{gf})/(P_m - P_{gi}) \right]$$

For valid results, the procedures given in 6.5.2, 6.5.3, and 6.5.4 must be followed, deleting any reference to getter specimens. The test chamber volume must be accurately known.

A3. MEASURING THE CONDUCTANCE TUBING BORE

A3.1   The radius *a* of the tubing used to fabricate a conductance can readily be determined using the mercury column technique. A weighed quantity of mercury is placed in the tubing and the length of the resulting mercury column accurately measured. Since

$$D = M/V = M/ (\pi a^2 l)$$

Then,

$$a = [M/ (\pi D l)]^{1/2}$$

where:
D = density, g/cm^3(kg/m^3),
M = mass of mercury, g (kg),
V = volume, cm^3(m^3),
a = radius, cm (m), and
l = length, cm (m).

The uniformity of the tubing bore is readily determined by locating the mercury column at several different positions in the tubing and measuring the variation in length.

F 798 – 97