

Reporting of Thermal Degassing Data

FOREWORD

This Foreword is not a part of AVS 9.2-1969. This publication specifies practices tentatively approved as standard by the American Vacuum Society so as to provide a consistent basis for the reporting of degassing measurements. It is one of a series published by the American Vacuum Society. It represents the best thinking of a number of experts in the field. After several years of use this standard will be forwarded to the USA Standards Institute with the request that it be used as a basis for a USA Standard. Suggestions for improvement gained in the use of this Standard will be welcome. They should be sent to the American Vacuum Society, 335 East 45 Street, New York, New York 10017.

This Standard was drafted by Dr. Peter Varadi.

The AVS Committees at the time of drafting and submission were as follows.

AVS Standards Committee

- D. G. Bills, Chairman, *Granville-Phillips Co.*
- B. B. Dayton, *Bendix Vacuum Division*
- A. Guthrie, *California State College-Hayward*
- A. Nerken, *Veeco Instruments, Inc.*
- G. Osterstrom, *Welch Scientific Co.*
- F. Reinath, *University of California*
- S. Ruthberg, *National Bureau of Standards*
- D. Stevenson, *Bendix Vacuum Division*
- P. Varadi, *Machlett Laboratories, Inc.*
- W. Wheeler, *Varian Associates*
- H. Schwarz, *Rensselaer Polytechnic Inst.*

AVS Subcommittee 9 Outgassing

- P. F. Varadi, Chairman, *Machlett Laboratories*
- M. F. Axler, *P.O. Box 5525, Grand Central Sta., New York, N. Y. 10017*
- C. P. Boebel, *RR1 BPOB 196A Brookville, Ohio 45309*
- R. H. Collins, *IBM Corp.*
- D. K. Das, *Raytheon Corp.*
- B. B. Dayton, *Bendix Vacuum Division*
- J. L. Lineweaver, *Corning Glass Works*
- M. Rivera, *Costa Mesa, California 92626*
- D. J. Santeler, *Aero Vac Corp., Box 448, Troy, New York 12181*
- R. A. Stehlow, *Oak Ridge National Lab.*
- S. A. Ward, *CBS Labs.*
- S. Dubuske, *U. S. Army Electronics*

1. SCOPE

This standard refers to the reporting of thermal degassing data. It is the objective of this standard to establish a consistent set of units, methods of data presentation, and certain experimental parameters in order that thermal degassing data from different sources may be useful to all. Nothing in the standard is intended to restrict the experimenter in any way.

2. UNITS

All units in degassing terminology will conform to the recommended standard units of the American Vacuum Society, specifically as follows:

Pressure	—Torr,
Gas Volume	—liter,
Time	—second in a compound unit, —second, minute, or hour for elapsed time,
Area	—square centimeter,
Temperature	—°Celsius or Kelvin,
Mass	—gram,
Sample Volume	—milliliter or cubic centimeter.

Compound units are to be written with a single slant bar or with negative exponents:

- Example (1) Torr liter sec cm²
- (2) Torr liter sec⁻¹ cm⁻².

Conversion factors to alternate sets of units are presented in Appendix B.

3. NOMENCLATURE

The following definitions of various degassing terms are recommended. (Use of the words "total" and "average" should be avoided unless properly clarified or defined.)

3.1. Thermal Degassing. Thermal degassing refers to deliberate removal of sorbed gases from a material by the application of heat transferred by radiation, conduction, and/or convection. This term should not be applied to degassing due to ion or electron bombardment, nor should it be confused with the terms "outgassing," "desorption," etc. (for clarification of these definitions see "Glossary of Terms Used in Vacuum Technology," issued by the Standards Committee of the AVS, Pergamon Press, Inc., New York).

3.2. Degassing Rate. Degassing rate is the quantity of gas (corrected to standard temperature) leaving a heated material per unit of time.

The experimenter has the choice of presenting the experimental data in

- (a) Torr liter/sec cm²
- (b) Torr liter/sec g,

according to whether it is desired to present the degassing data of the particular material as

- (a) a function of superficial surface (Torr liter/sec cm²)

or

- (b) a function of the bulk (Torr liter/sec g).

Unless otherwise stated, these units are referred to a standard temperature of 23° ± 3°C.

3.3. Evolved Gas quantity. Evolved gas quantity refers to the quantity of gas given off by a material between two stated times.

The experimenter has the choice of presenting the experimental data in:

- (a) Torr liter/cm²

or

- (b) Torr liter/g,

according to whether it is desired to present the evolved gas quantity of a material as

- (a) a function of the gross surface (Torr liter/cm²)
- (b) a function of the bulk (Torr liter/g).

Unless otherwise stated, these units are referred to a standard temperature of 23° ± 3°C.

3.4. Systems. If systems rather than materials are measured, where neither the surface nor the mass can be well defined, the data on degassing rate may be presented in Torr liter/sec and that for evolved gas quantity may be presented in Torr liters.

4. DATA PRESENTATION

Thermal degassing information may be presented in either graphical or tabular form or a combination of the two (see Sec. 4.1 and 4.2). Selection should be made on the basis of clarity of presentation.

To make reference to the text unnecessary, each data sheet should include sufficient identifying information of the sample, the sample temperature as a function of time, and the apparatus. A complete description of the sample, sample history, apparatus, and all other pertinent information should be supplied in an accompanying chart, appendix, or report (see Sec. 4.3 and 4.4).

4.1. Graphical Presentation. Any of the thermal de-

gassing information identified in Sec. 3.2 and 3.3 may be presented graphically as a function of time. In all cases the degassing data should be plotted on the Y (ordinate) axis and the complete identification and the units marked near the left margin. The time scale is plotted on the X (abscissa) axis in seconds, minutes, or hours, whichever is the most appropriate. The word "time" and the appropriate units are marked on the lower margin.

The preferred presentation is a log-log plot or a semi-log plot with time on the linear scale.

The number of curves per data sheet should be restricted to avoid multiple curve crossing or confusion.

When presenting thermal degassing data on materials that have been tested in a system, it is necessary to present thermal degassing and/or outgassing data of the empty system under similar test conditions (see 4.4.2). Pertinent information about the sample and the test apparatus should be given on the data sheet.

4.2. Tabular Presentation. Any of the thermal degassing information identified in Sec. 3.2 and 3.3 may be presented as a function of time in tabular form. In addition, data defined in Sec. 3.2 and 3.3 selected at a common time should be cross-referenced, in tabular form, against other identifying factors such as material type, area, pumping speed, etc., where the necessary information is available.

Where appropriate, the tabular data should include the thermal degassing information of the empty system.

4.3. Sample. A complete description of the sample material, component, or system, including identification, geometry, mass, and history should be given.

4.3.1. Identification. The sample should be completely identified by manufacturer, code or trade number, composition, density, age, or other factors which would be required for duplication of the same material.

4.3.2. Geometry and Mass. A complete description of the size, shape, exposed surface area, and mass of each sample should be supplied. In particular, a description of the surface roughness and finish should be supplied. (When convenient it is recommended that the ASA-B46.1-1962 Standard be used in describing the surface.)

4.3.3. History. The pertinent history of the sample prior to the test should be supplied in full detail. This history should contain the following information: age, storage conditions (temperature, time, humidity, environment), cleaning or chemical processing, handling, and prior degassing history.

4.4. Apparatus. In describing the apparatus used in a thermal degassing experiment, all information should be supplied which is required for duplication of the experimental results. The exact information required will vary with the nature of the test. The following terms are suggested and should be included whenever they are pertinent.

4.4.1. Vacuum System Speed. The reported vacuum pumping speed should be the summation of all pumping effects known to exist at the time of the experiment. Since some pumping effects are difficult to estimate, especially when the composition of the evolved gases is not known, it is desirable that the experimenter supply adequate descriptive information for duplication of the test results. Different system speeds, or changes in the pumping speed during the experiment or during initial exhaust, should likewise be reported in detail.

4.4.2. Background Degassing and/or Outgassing Rate. In those experiments involving a sample material in a system, the background degassing rate and/or outgassing rate should be small relative to the sample degassing rate. Information substantiating this should be supplied and may include comparative pressure-time data or comparative rate-of-rise data with the empty system and with the sample in place. In addition, the ultimate pressure of the empty system for the test conditions should be reported.

4.4.3. Time Measurement. Time measurements starts at the time when the sample heating begins. If sample outgassing is significant, then the time spent under vacuum prior to heating should be recorded as sample history (see Sec. 4.3).

4.4.4. Pressure Measurement. A complete description of the pressure measuring equipment should be supplied. This should include sensitivity, calibration, location, method of connection, and other descriptive factors.

If the gauge has been calibrated for a specific gas, such as nitrogen (or air), the degassing data should be reported as equivalent nitrogen (or air) degassing data. Wherever possible, the degassing data should be supplemented by partial pressure measurements. If the partial pressure data are the basic information, they should be converted to degassing data. If the partial pressures are supplementary, they may be reported as either partial pressures or as composition. In either event, the time at which the data were taken should be clearly indicated.

4.4.5. Temperature Measurement. In all instances, the temperature of the sample, the walls of the sample

chamber, and all other temperatures which may significantly affect the results and the measuring methods utilized should be stated. If any factors during the experiment change the sample or sample chamber temperature, the variations of temperature with time should be indicated. Information should be given on the method of heating the sample and the temperature of the sample as a function of time.

4.4.6. Miscellaneous Factors. The experimenter should report any and all factors that he feels are important to the experiment or significant in terms of duplicating his test results. Such factors may include sample suspension, system materials, etc.

APPENDIX A

A1. METHODS.

The intention of this standard is to provide a consistent set of units and nomenclature. Several methods currently used are described in the following sections, but it must be recognized that no one method is suitable for obtaining all thermal degassing data. It is the responsibility of the experimenter to choose the most suitable method for his particular application.

A1.1. Through-put Method. When a high-vacuum chamber is connected to a pumping system by an orifice or tube whose conductance is less than 5% of the speed of the pumping system, the net pumping speed in the chamber for the gas therein will be relatively independent of pressure and of variations in the speed of the pumps or efficiency of traps, provided that the pressure in the chamber is large relative to the ultimate pressure of the pumping system. While the net speed will be different for the various components in a gas mixture under molecular flow conditions, the throughput of each gas species leaving the chamber at any instant of time can be determined by measuring the partial pressure of the gas in the chamber and by multiplying by the net speed for that gas. The net speed can be computed from conductance formulae or measured by suitable calibration techniques. If the conductance of the orifice or tube is greater than 5% of the speed of the pumping system, the throughput may be determined by multiplying the conductance by the pressure difference across the orifice or tube. In the latter case, it is important that the temperature of the gas on the entrance side of the orifice be the same as the temperature on the exit side or else a suitable correction must be made.

A1.1.1. Degassing Rate Measurement. If the vacuum chamber is constructed of a material having a relatively low outgassing and/or degassing rate when properly cleaned or conditioned, then the degassing rate of a material may be determined by suspending

or supporting the sample of the material in the chamber, applying heat to the sample, and recording the pressure drop across the conductance. The degassing rate for a given gas is then calculated from the appropriate expression below:

$$296/T \cdot U \cdot (P_2 - P_1)/A$$

or

$$296/T \cdot U \cdot (P_2 - P_1),$$

where U is the conductance in liter sec⁻¹ of the orifice or a tube for the given gas at the temperature T prevailing at the entrance and exit, P_2 is the pressure in Torr for that gas on the entrance side of the orifice or a tube at the temperature T , P_1 is the pressure in Torr for that gas on the exit side of the orifice or tube at the temperature T (P_1 may be neglected if U is 5% or less of the speed of the pumping system as outlined in A1.1), A is the sample area in cm², G is the sample mass in grams, and T is the temperature in K of the gas on the entrance and exit sides of the orifice or tube (see A1.1).

If the background degassing and outgassing rate of the empty chamber and pressure measuring system are not negligible, then these values must be measured and accounted for by subtracting the background degassing and outgassing results from the results obtained while degassing a sample. Then, making background degassing and outgassing measurements, all experimental conditions should remain the same as for sample degassing measurements except for the absence of the sample. Also, care must be taken to insure that the background gases of the chamber do not influence the gases evolved from the material and that the pressure measuring equipment does not influence the types or quantities of gas one measures.

A1.1.2. Calculation of Evolved Gas Quantity. If the net pumping speed may be considered constant from time t_1 to time t_2 , then the evolved gas quantity can be obtained by integrating the pressure drop across the conductance as a function of time, multiplying it by the conductance, and dividing that quantity by the area or mass of the sample. The integration may be performed graphically when pressure is plotted vs time. A continuous record of data is preferred in order to observe abrupt changes in the gas evolution rate.

A1.2. Collection method. The obtaining of degassing data by the collection method involves pumping away the evolved gases from the heated sample by a diffusion pump and collecting the gases in a chamber of known volume. Mercury pumps are generally preferred in this application due to their greater tolerance in operating forepressure and the desire to avoid

vapors which are easily confused with the outgassing products being studied. At the end of a degassing period, which may vary from several minutes to many hours, the pressure of the gas or partial pressures of the gases is determined. The evolved gas quantity is then the product of the pressure and the volume of the collection chamber.

Information regarding degassing rate (Sec. 3.2) can be obtained by multiplying the slope of the pressure time curve by the calibrated volume when the pressure has been recorded continuously or at small time intervals, but the method is not accurate for slopes near zero.

As with the throughput method, background gases of the gas-analysis system, sorption of gases by the system, and changes in the original gas composition caused by the pressure measuring device, surfaces, etc., may influence the results. The experimenter must determine to what extent these effects may influence the results.

A1.3. Other Methods. Any method capable of supplying information required under this standard is applicable. Other methods in current use are described in Appendix A, A1, of Tentative Standard 9.1.

APPENDIX B

Conversion of Throughput in Torr Liters per Second to Particles per Second

$$N_p = N_a/R \cdot T \cdot Q$$

Q = throughput in Torr-liter per sec at temperature T

N_p = number of particles per second evolved from sample

N_a = Avogadro number = 6.02×10^{23} per g mole

R = gas constant = 62.36 Torr-liter K⁻¹ g mole⁻¹

T = absolute temperature of gas = 296 K (at standard room temperature)

$$N_p = 6.02 \times 10^{23} \cdot Q / 62.36 \times 296$$

$$N_p = 3.28 \times 10^{19} \cdot Q$$

APPENDIX C

SAMPLE DATA SHEET

Material	1	2
----------	---	---

1. Sample

- a. Identity
- b. Geometry (+ surface finish)
- c. Mass
- d. History
 1. Time/Temperature
 2. Processing
 3. Handling, etc.
 4. Miscellaneous

2. Apparatus

- a. Vacuum System Speed (4.4.1)
- b. System Outgassing Rate (4.4.2)
- c. Partial Pressure Measurement (4.4.4.)
- d. Temperature (4.4.5)
- e. Time

3. Degassing Rate (3.2)

- a. Surface (per cm²)

or b. Bulk (per g)

or c. System

4. Evolved Gas Quantity

- a. Surface (per cm²)
- b. Bulk (per g)
- c. System

5. Method used (also conversion factors)

6. Other Significant Factors