Designation: F 111 – 96 (Reapproved 2002)

Standard Practice for Determining Barium Yield, Getter Gas Content, and Getter Sorption Capacity for Barium Flash Getters[1]

This standard is issued under the fixed designation F 111; 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 describes techniques for the determination of evaporated barium yield, getter gas content, and getter carbon monoxide sorption capacity for barium flash getters used in electron devices. Test conditions are chosen to approximate use conditions.

1.2 Auxiliary procedures for cleaning, for determining vacuum system leak-up rates, for flashing getters, and for determining barium content in both getter fill and films are also given.

1.3 The various tests described are destructive in nature. In general the tests are semiquantitative but they can be expected to yield comparative information on a single-laboratory basis to the precision indicated. No information relative to multilaboratory reproducibility is available.

1.4 *List of Methods Described*:

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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 whoever uses this standard to consult and 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:*

D 1193 Specification for Reagent Water[2]

3. Terminology

3.1 *Definitions of Terms Specific to this Standard:*

3.1.1 *barium flash getters*—used to remove residual gases present after exhaust or generated during device operation by sorption with a barium film produced by heating the getter.

3.1.2 *barium yield, M*—the weight of barium in milligrams evaporated when a getter is flashed. Flash conditions are specified in terms of start time and total time.

3.1.2.1 *start time, t_s*—the interval in seconds between the application of heating power and the onset of barium evaporation. This value depends on the power applied.

3.1.2.2 *total time, t_t*—the full interval in seconds during which heating power is applied to the getter.

3.1.3 *carbon monoxide sorption capacity, C*—the quantity of CO sorbed at room temperature (25°C) measured in millitorr-litres until the terminal gettering rate is reached.

3.1.4 *conductance, F*—of a system for a given gas or vapor is the ratio of throughput of gas, Q, to the partial pressure difference across the system, $P_2 - P_1$, in the steady state. It is measured in liters per second, and given by $F = Q/(P_2 - P_1)$ where P_2 is the upstream pressure, and P_1 is the downstream pressure.

3.1.5 *flashing*—the evaporation of barium, contained within a getter, as a consequence of induction or resistance heating of the getter.

3.1.6 *Gas Content*:

3.1.6.1 *preflash gas content, PGC*—the quantity of gas in millitorr-litres reported as nitrogen equivalent evolved at flashing after it has been degassed at 350°C for 15 min under kinetic vacuum conditions.

3.1.6.2 *total gas content, TGC*—of a getter is the quantity of gas in millitorr-litres reported as nitrogen equivalent evolved at flashing when a getter is heated from room temperature.

[1] This practice is under the jurisdiction of ASTM Committee F01 on Electronics and is the direct responsibility of Subcommittee F01.03 on Metallic Materials.

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[2] *Annual Book of ASTM Standards,* Vol 11.01.

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

3.1.8 *gettering rate, G*—defined as the volume of gas sorbed in 1 s and is measured in litres per second.

3.1.8.1 *terminal getter rate*—reached when the gettering rate decreases to 1 L/s for large (TV) getters or 0.1 L/s for small (receiving tube) getters. It is used to define the end point of the CO sorption capacity test.

3.1.9 *getter test bulb*—that portion of the apparatus in which the getter is flashed (see Table 1 and Table 2).

3.1.10 *mass throughput, Q*—the quantity of gas flowing through a given plane in unit time and is measured in millitorr-litres per second.

3.1.11 *molecular flow region*—that pressure region where gases or vapors flow under conditions such that the largest internal dimension of a transverse section of the vessel is smaller than the mean free path. Under these conditions the rate of flow is limited not by collisions between molecules but by collisions of molecules with the walls.

3.1.12 *sorption by a getter*—the process of removing gases and vapors by adsorption and absorption phenomena.

3.1.12.1 *Absorption*, deals with gas interactions in the bulk of the getter film and is dependent on diffusion rates, solubility, and chemical reactions.

3.1.12.2 *adsorption*—describes gas interactions at the surface of the getter film.

3.1.12.3 Quantities of sorbed gas are measured in millitorr-litres.

TABLE 1 Suggested Bulb and Coil Dimensions

NOTE 1—The getters are centered in both coaxial and tangential coils.

Getter Type	Bulb			
	Number	Outside Diameter, mm	Length, mm	Height,[A] mm
Coaxial Flashing:				
Receiving tube	A	20	100	35
TV black and white	B	35	250	150
TV color	C	60	320	140
Tangential Flashing (optional):				
TV color	D	80	320[B]	90[C]

Getter Type	Coil			
	Number	Inside Diameter, mm	Height, mm	Turns
Coaxial Flashing:				
Receiving tube	E	23	16	3.5
TV black and white	F	51	22	5.5
TV color	G	70	22	5.5
Tangential Flashing (optional):				
TV color	H	51	15	6.0[D]

[A] Measured from bottom of getter to dome of bulb.
[B] Bulb axis inclined 30° from the vertical.
[C] Measured from center of getter to dome of bulb.
[D] A two-layer coil with three turns per layer.

TABLE 2 Bulbs and Coils for Determination of CO Sorption Characteristics

Getter Type	Test Bulb	Test Bulb Dimensions	Coil
Receiving tube (all)	spherical bulb	100-cm^3 spherical flask (OD 60 mm)	F
OD 12–13 mm TV	standard TV bulb	12-in., 110° neck, OD 20 mm	E
OD 18–19 mm TV	standard TV bulb	19-in., 114° neck, OD 28.5 mm	F
OD 25–26 mm TV	standard TV bulb	23-in., 92° neck, OD 36.5 mm	F H[A]

[A] Used for antenna-mounted getters.

4. Safety Hazards

4.1 Eye protection is mandatory in the presence of large evacuated glass vessels or picture-tube bulbs, which should also be surrounded by suitable mechanical protection against implosion.

5. Test Specimens

5.1 The test specimens are commercial barium flass getters used in electron devices. The major components of a getter are: the fill, the container, and the support.

5.1.1 *The Getter Fill* is based on barium alloy, $BaAl_4$, to minimize the reactions of barium with the atmosphere.

5.1.1.1 An endothermic getter fill uses the $BaAl_4$ alloy alone and requires continued heating above 1050°C to liberate and evaporate the barium.

5.1.1.2 An exothermic getter fill is produced by intimately mixing about four moles of nickel with one mole of the barium alloy. On heating to about 800°C, the nickel reacts exothermally with the alloy, liberating and evaporating 10 to 30 % of the barium. Continued heating is required to evaporate most of the remaining barium.

5.1.1.3 The getter may be gas doped. Hydrogen may be added to the fill for reported beneficial effects on electron emission. Nitrogen may be added to influence the distribution and to produce a more porous film and thereby increase the gas sorption capacity as measured below.

5.1.2 *The Getter Container* form depends on the method of supplying heating power for flashing.

5.1.2.1 If resistance heating is to be used it should have an open geometry, more or less rectilinear.

5.1.2.2 If induction heating is to be used it should have the form of a closed loop. The present trend is to employ a ring-shaped channel. In such cases the getter-channel cross section may be varied to influence, to a certain extent, the getter film-deposition pattern.

5.1.3 *A Getter Support* is used for mounting and positioning the getter in electron devices. It may or may not be an integral part of the getter.

5.2 Nominal getter sizes which are currently available are listed in Appendix X1.

5.3 *Getter Description*—The getter manufacturer shall furnish on request the following data:

5.3.1 Type of fill (endothermic or exothermic).

5.3.2 Recommended yield, start, and total times.

5.3.3 If gas doped or not. If doped, then with which gas; and the maximum temperature time condition which causes no loss of doping.

5.3.4 Container size (outside diameter in millimetres for ring getters; length times width, each in millimetres, for loop getters; and length, in millimetres for resistance-heated getters).

5.3.5 Container (channel) shape (ring with high inside wall, ring with high outside wall, ring with equal height walls, getter tubing, and so forth).

5.3.6 If magnetic or antimagnetic.

5.3.7 Shape of support (ribbon, wire, tab).

5.3.8 Special features such as ceramic spacers, etc., if any.

5.4 *Getter Lot Number*—To any getter production batch a lot number is assigned and the production date is given. From the lot number, date, and the internal manufacturer's control charts it shall be possible to trace back all the production cycles up to the incoming raw materials.

5.5 *Specimen Handling*:

5.5.1 Handle getters only with clean tools, lint free gloves, or finger cots, never with bare hands.

5.5.2 For long term storage, store the getters in a phosphorus pentoxide air desiccator or equivalent. As soon as the getter can is opened, place getters to be tested in a conventional silica gel desiccator. Initiate all measurement between 24 and 48 h after placing the getters in the conventional silica gel desiccator.

6. Cleaning Procedures

6.1 *Scope*—The following cleaning procedures shall be used for getter mounts and getter test bulbs used in Sections 10, 11, and 12.

6.2 *Significance*—Cleaning procedures are necessary not only to ready the mount and bulb for the tests but also to minimize possible errors. A residue of barium can cause errors in the barium yield determination when chemical methods are used. Oils and greases can adversely effect the vacuum.

6.3 *Materials and Reagents*:

6.3.1 *Hydrochloric Acid* (1 + 17)—add 1 part of concentrated HCl (sp gr 1.19) to 17 parts of deionized water.

6.3.2 *Deionized Water* (*DIW*)—At least 2-MΩ resistivity.

6.3.3 *Acetone, cp*.

6.3.4 *Cleaning Solution*—Add 2 volumes of concentrated HF (sp gr 1.15), 33 volumes of concentrated HNO_3 (sp gr 1.42), and 2 volumes of surface active agent to 100 volumes of DIW.

6.3.5 Alkylaryl polyether alcohol (OPE—7 to 8) is a liquid, nonionic surface active (wetting) agent. It is listed as an industrial detergent and emulsifier, effective in aqueous mineral acids with good hard (glass) surface detergency and low foam. Any such agent, without filler, ionic or nonionic that meets the properties listed above is acceptable.

6.4 *Mount Cleaning*:

6.4.1 Wash with HCl (1 + 17).

6.4.2 Rinse with DIW.

6.4.3 Dry with acetone.

6.4.4 Handle only with clean tools, lint free gloves, or finger cots.

6.5 *Getter Test Bulb Cleaning*:

6.5.1 Wash twice with HCl (1 + 17) to dissolve barium.

6.5.2 Rinse with tap water.

6.5.3 Rinse with acetone to eliminate oils and greases.

NOTE 1—The acetone may be replaced by the more active cleaning solution of 6.3.4. Use solution at room temperature.

6.5.4 Rinse twice with DIW.

6.5.5 Drain.

6.5.6 Dry using a clean oil-free air blast, or better: vacuum dry, with or without bake, using a trapped mechanical pump to minimize hydrocarbon contamination.

7. Leak-Up Rates

7.1 *Significance*—The leak-up rate gives a measure of the cleanliness and freedom from leaks of vacuum system.

7.2 *Summary*—The vacuum test chamber which contains a pressure gage is isolated from the vacuum pumps by a suitable valve and the changes in pressure with time are recorded to obtain the data needed to calculate leak-up rates. A gas burst may be observed initially on closing the valve due to the liberation of sorbed gases in the valve, but the pressure will reach a steady-state value within several seconds. The pressure may then continue to rise in a manner controlled by any real leaks and relatively high vapor pressure contaminants within the vacuum chamber. The pressure may decrease with time indicating a clean leak-free system and pressure-gage pumping.

7.3 *Procedure*:

7.3.1 Measure the leak-up rate during the determinations of gas content (11.4.4) and sorption capacity (12.4.7). The apparatus used is described in these sections. At the appropriate stage of these determinations, indicated in the relevant text, valve off the test chamber from the vacuum pumps and record the initial pressure P_i. After a time, t, record the final pressure P_f.

7.3.2 The value of P_f and P_i should be such that there is a reasonable difference between them. Allow a change of at least a factor of 2 in pressure unless the leak-up rate is extremely low.

7.4 *Calculations*—Calculate the system leak-up rate, Q_L, in millitorr-litres per second using the following equation:

$$Q_L = V(P_f - P_i)/t$$

where:

V = system volume, litres,
P_f = final pressure, mtorr, in the valved-off test chamber,
P_i = initial pressure, mtorr, in the valved-off chamber, and
t = time of pressure rise, s.

7.5 *Sensitivity*—Using commercial Bayert-Alpert design ionization gages and controls (10.10^{-9} torr most sensitive full-scale range) and a 1-L volume leak-up rates of 1.10^{-8} mtorr·litres/s are readily measurable.

8. Getter Flashing

8.1 *Summary of Procedure*—Getters are flashed by induction or resistance heating using the required power to achieve the specified start time. This power is applied for the specified total time. The quantity of barium evaporated can then be determined using the analytic methods given in Section 9.

8.2 *Significance*—The barium yield of a getter when flashed at the manufacturer's recommended start and total times is one of the factors considered in selecting a particular getter for a specific application. The variation in barium yield caused by changes in start and total times are of interest to define the effects of application and production variables.

8.2.1 It should be noted that induction heating is not a simple phenomenon. The generator design, the frequency used, the reflected load, flashing techniques and procedures, etc. can influence the yield.

8.3 *Apparatus*:

8.3.1 *Induction Heater* (frequency must be between 250 and 450 kHz) or resistance heating apparatus.

8.3.2 *Bulb and Work Coils* (Table 1).

8.3.3 *Stop Watch*.

8.3.4 *Preset Timer*.

8.4 *Procedure*:

8.4.1 Position work coils as specified in the footnote to Table 1, and with reference to Table 3.

8.4.2 Set power input into heater for desired start time.

8.4.3 Start heating and timing.

8.4.4 Measure start time to an accuracy of 0.5 s or better using the stop watch.

8.4.4.1 The onset of barium evaporation is indicated for endothermic getters by the appearance of a film on the bulb.

8.4.4.2 The onset of barium evaporation is indicated for exothermic getters by the sudden temperature increase visually observable on the surface of the fill.

8.4.4.3 In both cases a sudden pressure drop will be observed.

8.4.5 Control total time by a preset timer to an accuracy of 0.5 s or better.

8.4.6 During the getter flash cycle watch the getter for uniformity of heating, evidence of loose particles, and type of gas discharge, if any.

8.4.7 Use resistance heating for getters that are not of closed shape, for example wire getters. Connect the ends of the getter to an alternating- or direct-current source via a suitable ballast resistor. The ballast resistor must have a resistance 20 to 30 times greater than that of the cold getter and have a power rating sufficiently large to support the current required to flash the getter. The resistance of the current feedthrough, legs, and mounting support shall be small compared to the rest of the circuit. Connect an ammeter in series with the getter and use it to determine the power supplied. Connect a voltmeter in parallel with the current feedthroughs to evaluate the variation of getter resistance.

TABLE 3 Getter Positions

Getter Type	Test Bulb	Getter Position
Receiving tube (all)	spherical bulb	at the neck position with its plane tangential to the projected spherical surface of the bulb
OD 12–13 mm TV	standard TV bulb	27.0 mm from *YRL* coaxial with neck
OD 18–19 mm TV	standard TV bulb	27.0 mm from *YRL* coaxial with neck
OD 25–26 mm neck-mounted	standard TV bulb	41.3 mm from *YRL* coaxial with neck
OD 25–26 mm antenna-mounted TV	standard TV bulb	The center of the getter is 22 mm from *YRL*. The plane of the getter is parallel to the surface of the funnel.

9. Barium Determination

9.1 *Scope*—Chemical and physical analytical procedures are given for the determination of barium in the getter-alloy fill and in the evaporated barium film. The procedures to be described are listed in Table 4. Composite apparatus, materials, and reagent lists are given. Summaries, procedure, calculation and accuracy are given under each method.

9.2 *Reagents and Materials (General Recommendations)*:

9.2.1 *Purity of Reagents*—Reagent grade chemicals will be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society or other recognized institution where such specifications are available.[3]Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

9.2.2 *Purity of Water*—Unless otherwise indicated, reference to water shall be understood to mean reagent water conforming to Specifications D 1193.

9.3 *Weight Difference*:

9.3.1 *Principle of Method*—The getter and its support leg is weighed before and after getter flash. The weight difference is the quantity of barium evaporated. This method is a very rapid, accurate, and simple method for determining the yield if the getter is not overflashed. It is the recommended method for barium yields greater than about 10 mg.

9.3.2 *Apparatus*:

9.3.2.1 *Analytical Balance*, 0.1 mg sensitivity or better.

9.3.2.2 *Apparatus for Getter Flash*, as described in 8.3.

9.3.3 *Procedure*—Weigh the getter accurately to the nearest 0.1 mg. Mount the getter, flash, and reweigh. The flashed getter should be reweighed within 15 min after exposure to air. Overflashing, as evidenced by channel burn and loose particles, can affect the measurement.

9.3.4 *Calculations*—The weight difference before and after flash is the amount of barium evaporated.

9.3.5 *Accuracy*—The method is accurate to ±0.5 %.

[3] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharacopeial Convention, Inc. (USPC), Rockville, MD.

TABLE 4 Methods for Determination of Barium

Method		For Barium in		Mass of Barium, mg
		Fill	Film	
Weight difference	(9.3)		x	>2
Gravimetric	(9.4)	x		>50
Spectrophotometric	(9.5)	x	x	all

9.4 *Gravimetric Determination of Barium in the Fill*:

9.4.1 *Summary of Method*—Barium is precipitated from solution as barium sulphate and a direct weighing of the precipitate is made.

9.4.2 *Reagents and Materials*:

9.4.2.1 *Hydrochloric Acid (1 + 1)*—Mix equal volumes of concentrated HCl and water.

9.4.2.2 *Sulfuric Acid (1 + 200)*—Add 5 mL of concentrated H_2SO_4(density 1.82 g/mL) to 1 l of deionized water.

9.4.2.3 *Litmus Paper*.

9.4.3 *Procedure*—Dissolve 250 mg of endothermic or 500 mg of exothermic-getter alloy from one or more getters in the smallest possible amount of HCl (1 + 1). Evaporate nearly to dryness and then dilute with water to between 200 and 300 mL. Heat the slightly acid (HCl) solution of $BaCl_2$ to boiling and add a slight excess of H_2SO_4(1 + 200). Settle the precipitate on the water bath and decant the clear solution through a weighed Gooch crucible or through an ashless filter paper. Transfer the precipitate to the crucible (or paper) and wash twice with H_2SO_4(1 + 200), and finally with hot water until free of acid. Dry and ignite the precipitate at first gently and then over a good flame to a cherry-red heat, for 30 min. Weight the residue as barium sulphate, $BaSO_4$, to the nearest 0.1 mg.

9.4.4 *Calculations*—Calculate the weight of barium as follows:

$$\text{Barium, mg} = 0.586\,A$$

where:

A = weight of $BaSO_4$ in mg.

9.4.5 *Accuracy*—This method is accurate to ±0.5 %.

9.5 *Spectrophotometric Method*:

9.5.1 *Principle of Method*—The powder extracted from the getter container is dissolved in HCl and the barium concentration is determined by means of an atomic absorption spectrophotometer.

9.5.2 *Apparatus*:

9.5.2.1 *Analytical Balance*, 0.1 mg sensitivity or better.

9.5.2.2 *Atomic Absorption Spectrophotometer*.

9.5.2.3 *Microburet*.

9.5.3 *Reagents and Materials*:

9.5.3.1 *Barium Solution*, certified standard, 10 000 ppm.

9.5.3.2 *Lanthanum Chloride*.

9.5.3.3 *Hydrochloric Acid (1 + 1)*—Mix equal volumes of concentrated HCl and water.

9.5.4 *Procedure*:

9.5.4.1 *Preparation of 10 % Lanthanum Chloride Solution*—Weight 100 g of lanthanum chloride and dissolve in 1 l of deionized water.

9.5.4.2 *Preparation of Barium Standards*—Draw aliquots of 0.0, 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 mL from the 10 000 ppm barium standard solution and decant them in 100-mL volumetric flasks. Add 2.5 mL of HCl (1 + 1) and 5 mL of lanthanum chloride 10 % solution to any flask. Make up the volume with deionized water and shake. The standard solutions thus prepared have a barium concentration respectively of 0 (blank), 50, 100, 150, 200, 250, 300 ppm.

9.5.4.3 *Preparation of the Sample Solution*:

(*1*) Weight the powder extracted from the getter container to the nearest 0.1 mg and pour it in a 250-cm^3 flask. Slowly add 30 mL of HCl (1 + 1) and then heat moderately until all the sample has been dissolved. Continue heating to concentrate the solution down to about 10 mL for samples of more than 200 mg and nearly to dryness for samples of less than 200 mg.

(*2*) Cool to room temperature and decant the liquid into suitable volumetric flask and make up the volume with the wash water and water, ensuring a complete transfer of the sample.

(*3*) Prepare a final solution, eventually through successive dilution, so as to obtain a barium concentration ranging from 100 to 200 ppm.

(*4*) Before filling the final volumetric flask, add the 10 % lanthanum solution and HCl (1 + 1) to the drawn aliquots. The volumes to be added are respectively of 5 mL and 2 mL, referring to a final volume of 100 mL. When adding HCl take into account the eventual residual volume of the sample after drying.

9.5.4.4 *Measurement*:

(*a*) Set the atomic absorption spectrophotometer to the following conditions:

(*1*) Wavelength: 553.6 nm,

(*2*) Slit Aperture: 0.14 nm,

(*3*) Flame: air/acetylene, in slightly reducing conditions.

(*b*) Set burner and lamp current according to the directions of the manufacturer.

(*c*) Suggested reading time is 7 s. Number of readings to be averaged should be three.

(*d*) Set zero by means of the blank solution and read the standard solutions twice. Ensure deionized water is admitted in the atomic absorption spectrophotometer for a sufficient time before analyzing a new standard.

(*e*) Perform analyses on the samples.

(*f*) Repeat analyses on the standard solutions and compare the results with the previous ones.

(*g*) The measurements on the actual samples can be considered acceptable if the readings on the standard solutions, before and after the measurements on the samples, do not differ of more than 1 %.

9.5.5 *Calculations*:

9.5.5.1 Calculate the amount of barium as follows:

$$\text{Barium, mg} = \frac{A \cdot B \cdot 100}{1000}$$

where:

A = ppm of barium, and

B = dilution factor.

9.5.5.2 Calculate the percentage of barium as follows:

$$\text{Barium, \%} = \frac{\frac{A \cdot B \cdot 100}{1000}}{W} \times 100$$

where:

A = ppm of barium,

B = dilution factor, and

W = weight of the sample in mg.

9.5.6 *Accuracy*—This method is accurate to ±0.2 %.

10. Determination of Barium Yield

10.1 *Summary*—Flash getters when heated to a sufficiently high temperature evaporate a quantity of barium that is a function of the start time and the total time. To describe the barium yield properties for each type of getter one or more yield curves are plotted which give the mass of barium evaporated as a function of the start time with the total time as a parameter.

10.2 *Significance*—One of the factors in selecting a barium flash getter is the available barium yield for specified start and total times. Since these times vary in use due to the specific application and process variables, the changes in yield with these parameters is evaluated. The barium yield may be compared to the total amount of barium in the getter-alloy fill.

10.3 *Apparatus*:

10.3.1 The test apparatus is shown schematically in Fig. 1.

10.3.2 The pumping system must have the following characteristics:

10.3.2.1 An ultimate vacuum of less than 5.10^{-4} Torr and

10.3.2.2 Adequate pumping speed so that the maximum pressure during flashing is less than 0.1 Torr.

10.3.3 Suggested cylindrical bulb dimensions, getter mounting position (from the base of the getter to the dome of the bulb), and work coil dimensions are given in Table 1. The getter is centered in both the cylindrical and tangential coils. Normally the getters are flashed with a coaxial coil in the horizontal position with the flash directed toward the dome of the bulb. For antenna mounted color TV getters, a tangential coil may be used to cause flashing towards the opposite wall. In this case, the getter mount should be at an angle of at least 30° to the vertical to prevent motion of the getter during flash. The getter must be placed at a suitable distance from major metal parts to prevent rf losses. The use of a spoiler strip to prevent rf losses due to the continuous barium film is optional.

10.3.3.1 A typical apparatus for wire getters is shown in Fig. 2. The wire getter is parallel to the axis of the tube and should flash towards the nearest wall.

10.3.4 The pressure gage, G_1, must measure pressures ranging from 5.10^{-4} to 0.1 Torr.

10.4 *Procedure*:

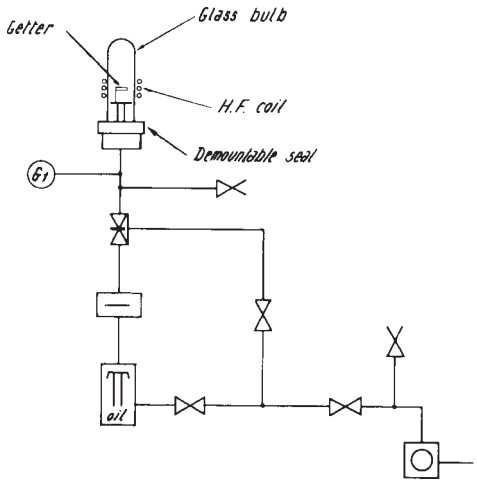


FIG. 1 Schematic of Barium Yield Determination Apparatus

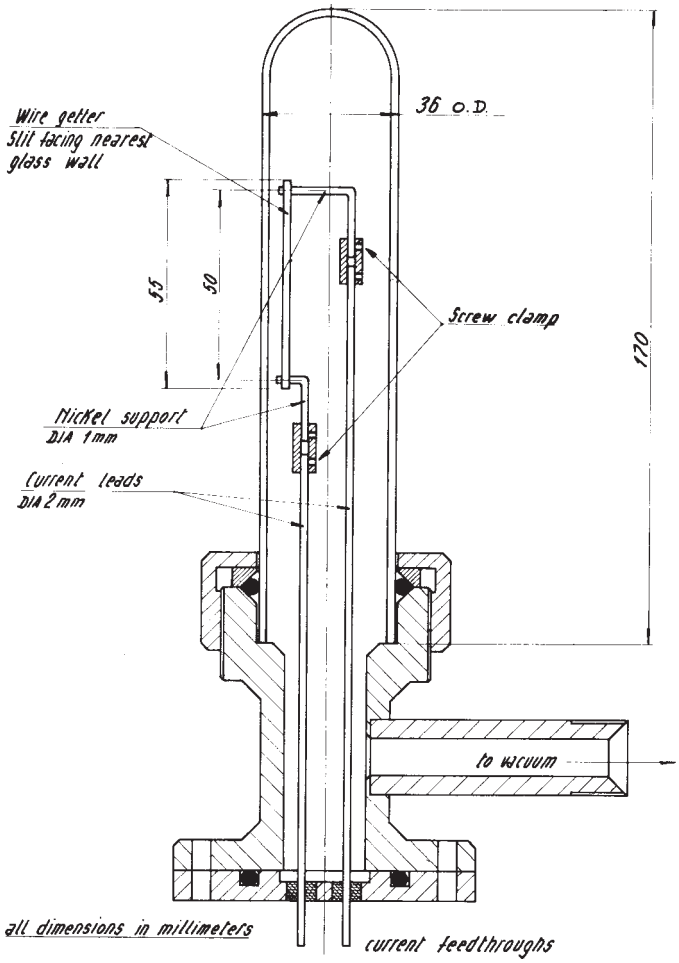


FIG. 2 Getter Mounting for Yield Determination: Wire Getters

10.4.1 Observe the clean handling techniques of 5.5.1 for both getter and mount.

10.4.2 Weigh the getter with its integral support. Sections 10.4 and 12.4 describe only the use of the weight difference method of determining the barium yield. Any of the chemical methods, given in Section 9, can be used. In these cases, the weighing of the getter before and after flash is not required and the yield calculations are those of the relevant part of Section 9.

10.4.3 Attach test getter to its appropriate cleaned mount.

10.4.4 Secure the mounted getter in the test apparatus.

10.4.5 Evacuate the system to 5.10^{-4} Torr or less.

10.4.6 Flash the getter by applying the necessary heating power. Record both the start and total times.

10.4.7 Admit air into the system; remove the glass bulb and the getter with its integral support. Check that there is no fusion of the getter container.

10.4.8 Weight the getter with support.

10.4.9 Determine the average weight, $\bar{M}_f$, in milligrams of barium in the fill using the procedures given in 9.4 or 9.7 and a minimum sample of ten getters.

10.4.10 Flash n getters, where n is greater than 9, in sequence at constant total time and constant applied power. The power and the total times are such that the manufacturer's

suggested start and total times are used in this determination of consistency of getter yield.

10.4.10.1 Note that consistency of yield not only indicates getter variations but also variations produced in the flashing assembly and thus gives a measure of the barium-yield variations which may be encountered in the production use of getters.

10.4.11 To obtain the data for the barium yield curves, flash *n* getters, where *n* is greater than 9, at constant total time and variable start time. The start time, determined by the applied power, should begin at the start-time value at which there is the slightest indication of fusion of the getter container and proceed to the start-time value at which the barium yield is 30 % of the manufacturer's recommended value. Repeat these measurements for two other total times.

10.5 *Calculations*:

10.5.1 *The Barium Yield, M_y*, in milligrams, is given by the following equation:

$$M_y = W_i - W_f$$

where:

W_i = initial weight of the getter, mg, and

W_f = weight of getter, mg, after flashing if there is no overflashing of the getter (see section 9.8.3.)

10.5.2 *The Percent Barium Yield*, percent Ba, is given by the following equation:

$$\text{percent Barium} = 100\ (M_y / \bar{M}_f)$$

where:

M_y = barium yield, mg, and

$\bar{M}_f$ = average weight of barium in the fill, mg.

10.5.3 *Consistency of Yield*—Using the results of 10.4.10 compute the average barium yield, $\bar{M}_y$, and average start time, $\bar{t}_s$, and their standard deviation, σ.

10.5.4 *Barium Yield Curve*—Plot the barium yield curve using the data of 10.4.11 by reporting on a linear scale the start time in seconds as the abscissa and the yield, in milligrams, as ordinate, for a given total time. The ordinate covers the range of practical use which is recommended by the manufacturer. Generally three curves, having different total times as the parameter, are presented. These curves should also show the following:

10.5.4.1 Manufacturer's standard getter nomenclature,

10.5.4.2 Average weight of alloy fill in milligrams and its standard deviation,

10.5.4.3 Whether fill is endothermic or exothermic,

10.5.4.4 Type of gas doping if any,

10.5.4.5 Bulb, coil type, and flashing frequency used.

10.6 *Report*:

10.6.1 The barium yield report consists of:

10.6.1.1 Barium yield,

10.6.1.2 Percent barium yield,

10.6.1.3 Consistency of yield date (see 10.5.3), and

10.6.1.4 Barium yield curve.

10.6.2 The yield data for 10.6.1.1, 10.6.1.2, and 10.6.1.3 are measured at the manufacturer's recommended start and total times. Report the number of samples and the standard deviation. The report will be referenced in terms of the manufacturer's standard getter nomenclature and lot numbers will be given where applicable. It is suggested that these data be reported on the barium yield curve.

10.7 *Precision*—The expected precision is ±5 %.

11. Determination of Getter-Gas Content, Total and Preflash

11.1 *Summary*:

11.1.1 The getter-gas content is determined by heating the getter to its flashing temperature in a closed system of known volume and measuring the maximum pressure attained, which occurs just prior to getter flash. For gas-doped getters, the total quantity of hydrogen alone can be determined physically and the total quantity of nitrogen, chemically.

11.1.2 The getter-gas content determination can be effected by taking the getter directly from the ambient temperature of the flashing temperature, giving the measure of total gases, *TGC*. If, however, the measurement is made by preheating the getter at 350°C for 15 min under diffusion pumping, then the measure of preflash gas, *PGC*, will be obtained. The latter test is preferred for TV getters as it simulates working conditions removing physisorbed gases which are normally removed during exhaust-baking production processes.

11.1.3 Useful information about alloy stability can be obtained if the total and preflash gas tests are also performed after a 2-week exposure to ambient room air. If comparison tests are being made all samples must have identical exposure.

11.1.4 In order to obtain reproducible results the following start times are recommended: receiving tube getters, 10 ± 2 s; TV getters black and white, 15 ± 2 s; and TV getters color, 25 ± 2 s.

11.1.5 The start time with which the getters may be flashed and still give constant results can vary over rather wide limits but the minimum start time will be limited by the rate of response of the gage tube and its associated circuitry. It is usual practice to use start times longer (and hence lower flashing power) than those used for the barium yield determinations; therefore, the two measurements of barium yield and gas content cannot be obtained from the same sample.

11.2 *Significance*—For gas-doped getters, the amount of gas added and its variation is important in achieving the desired results. The amount and variability of gas present in nondoped getters is useful in quality control. Sorption of atmospheric gases or contamination by improper handling, or both, can be sources of error.

11.3 *Apparatus*:

11.3.1 The test apparatus is shown schematically in Fig. 3. It consists of a known volume capable of being isolated from the pumping system in which the getter is flashed.

11.3.2 The ultimate pressure must be 5.10^{-5} Torr or lower and the leak-up rate must be less than 1.10^{-2} mtorr·litre/s.

11.3.2.1 The total volume of the system varies according to the type of getter under test. Recommended volumes are as follows:

Receiving tube getters	0.5 to 1 L
TV getters black and white	1 to 2 L
TV getters black and white doped	5 to 6 L
TV getters color	5 to 6 L
TV getters color doped	18 to 20 L

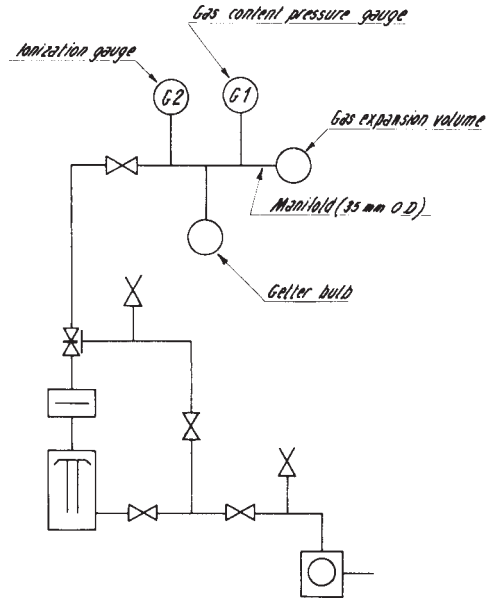


FIG. 3 Schematic of Gas Content Determination Apparatus

The use of these volumes will keep the peak pressure in the range of greatest accuracy for available instrumentation.

11.3.2.2 Determine the system volume to an accuracy of 5 % or better by calculation or by determining the quantity of water required to fill the apparatus.

11.3.3 Refer to Table 1 and 10.3.3 for bulb and coil data.

11.3.3.1 Preflash gas measurements require a getter temperature sensing device. The use of a thermocouple is convenient. This can be in the form of two supports one being Alumel and the other Chromel, thus forming a contact thermocouple.

11.3.4 Ionization gage, G_1, can be any conventional ionization gage. The gas content pressure gage, G_2, must: read accurately in the 0.01 to 0.1-Torr range, have a response time of less than 0.5 s, and possess a stable calibration with time and repeated air exposure.

11.3.4.1 A strip chart recorder may be attached to the G_2 control unit. The recorder must have a full-scale response time of less than 0.5 s and a chart speed in the range of 4 to 8 in./min. The gas content pressure gage is calibrated against an absolute McLeod gage using nitrogen.

11.4 *Procedure for Total Gas Content*:

11.4.1 Observe the clean handling techniques of 5.5.1.

11.4.2 Attach test getter to its appropriate cleaned mount.

11.4.3 Secure the mounted getter in the test apparatus.

11.4.4 When the pressure is below 5.10^{-5} Torr, close the isolation valve and measure the leak-up rate (see 7.4). If less than 0.01 mtorr·L/s, repump to below 5.10^{-5} Torr.

11.4.5 Close the isolation valve and flash the getter (see Section 8) using the start times of 11.1.4. Measure the pressure changes during the flash cycle. Be sure to record the peak pressure.

11.4.6 Admit air into the system.

11.4.7 Mechanically remove the getter-alloy fill by distorting the container from 9 or more getters. Weigh the fill from each getter to an accuracy of 0.1 mg.

11.5 *Procedure for Preflash Gas Content*:

11.5.1 Observe the clean handling techniques of 5.5.1.

11.5.2 Attach the getter to the thermocouple leads of the appropriate cleaned mount so that flashing is directed towards the dome of the test bulb.

11.5.3 Secure the mounted getter in the test apparatus.

11.5.4 When the pressure is below 5.10^{-5} Torr measure the leak-up rate, if less than 0.01 μmtorr·litre/s, repump to below 5.10^{-5} Torr. Then heat the getter to 350°C in 20 to 30 s. Determine the temperature using the thermocouple or any other convenient means. Maintain the getter under pumping at this temperature for 15 min. Remove the heating power and let the getter cool down until the temperature is just below 100°C.

11.5.5 Close the isolation valve and flash the getter at the desired start time (11.1.4). Measure the pressure changes during the flash cycle. Be sure to record the peak pressure.

11.5.6 Admit air into the system.

11.5.7 Mechanically remove the getter-alloy fill by distorting the container from 9 or more getters. Weigh the fill from each getter to an accuracy of 0.1 mg.

11.6 *Calculation of TGC and PGC*:

11.6.1 Total gas content in millitorr-litres is given by the following equation:

$$TGC = V(P_f - P_i) - Q_L t_s$$

where:

P_f = observed peak pressure, mtorr,
P_i = initial pressure, mtorr,
V = system volume, Ls,
Q_L = system leak-up rate, mtorr·litres/s, and
t_s = start time, s.

The equation is identical for *PGC*. Generally both P_i and $Q_L t_s$ are negligible, in which case the above equation reduced to:

$$TGC \text{ or } PGC = VP_f \text{ mtorr·litres.}$$

11.6.2 Gas content per milligram of getter-alloy fill, γ, is calculated by the following equation:

$$\gamma_t \text{ or } \gamma_p = GC/W$$

where:

GC = gas content, total or preflash, mtorr·litres and
W = average weight of the getter-alloy fill, mg.

11.7 *Determination of Hydrogen Content of Hydrogen Doped Getters*:

11.7.1 *Principle*—The gas to be analyzed is brought into contact with a heated palladium tube where the hydrogen diffuses through leaving behind all other gases.

11.7.2 *Apparatus*—A palladium tube is attached to the manifold of the gas content apparatus of Fig. 3. An isolation valve is attached between this tube and the manifold to isolate the palladium tube during gas evolution and prevent any loss of hydrogen before the peak pressure is read.

11.7.3 *Procedure*—Follow the procedure given in 11.4 or 11.5. Keep the palladium tube portion open during system pump down but close it prior to getter-gas evolution. (**Warning**—Do not flash the getter during gas evolution.) Instead bring the getter to a dull-red heat (about 700°C for both endo- and exothermic getters) and maintain this temperature until the evolution of gas ceases (less than 5 min). Record this

pressure. Open the isolation valve to the palladium tube. Initially the pressure drops rapidly due to the expansion of gas into the tube volume and continues to drop until most of the hydrogen has diffused out of the system. Record the final pressure, P_R. Calibrate the gas-content pressure gage periodically against an absolute McLeod gage for both hydrogen and nitrogen.

11.7.4 *Calculations*:

11.7.4.1 The expanded gas pressure is calculated in terms of millitorr hydrogen equivalent by the following equation:

$$P_{ex} = PV_1/(V_1 + V_2)$$

where:

P_{ex} = pressure of the expanded gas (H_2), mtorr,
P = maximum pressure before expansion, hydrogen equivalent, mtorr,
V_1 = system volume less the palladium tube portion, litres, and
V_2 = volume of the palladium tube portion, litres.

11.7.4.2 *The Hydrogen Gas Content, HGC*, in millitorr-litres is given by the following equation:

$$HGC = P_{ex}(V_1 + V_2) - P_R(V_1 + V_2) - Q_L t$$

where:

P_R = pressure of residual gas, mtorr, nitrogen equivalent (the final pressure),
Q_L = system leak-up rate, mtorr·litres/s, N_2, and
t = test time, s.

Due to the length of time necessary to complete this analysis, the leak-up rate term can be significant. Residual hydrocarbons, water, etc. may be cracked releasing hydrogen which contributes to the measured hydrogen content of the getter.

11.7.5 *Precision*—The precision is ±10 %.

11.8 *Determination of Nitrogen Content of Nitrogen-Doped Getters*:

11.8.1 *Summary*—The sample is digested with sulfuric acid to convert the nitrogen into $(NH_4)_2SO_4$. The solution is then made alkaline with NaOH to form NH_3 which is removed from the digest by distillation and determined by titration.

11.8.2 *Apparatus*—The apparatus consists of a 500-cm^3 Kjeldahl flask furnished with a small glass tube for passing a stream of inert gas (argon) through the solution during distillation. An electric heater is placed around the bulb. The flask is provided with joints for installation of a 500-cm^3 separatory funnel and a condenser (these parts are attached after the digestion is completed). The condenser is connected to a special absorption bottle furnished with a buret for titration in the same vessel (Fig. 4).

11.8.3 *Reagents* (see 9.3.1 and 9.3.2).

11.8.3.1 *Sulfuric-Phosphoric Acid Mixture*—To 1 L of water add slowly, while stirring, 300 cm^3 of concentrated sulfuric acid. Cool and add 90 cm^3 of phosphoric acid. The mixture shall be prepared in an atmosphere free of ammonia fumes.

11.8.3.2 *Sodium Hydroxide Solution* (333 g/L)—Dissolve 333 g of NaOH in 1 L of water.

11.8.3.3 *Indicator Solution*—Dissolve 30 mg of bromocresol green and 20 mg of methyl red in 50 cm^3 of methanol. This indicator changes from wine-red to green at pH 5.1.

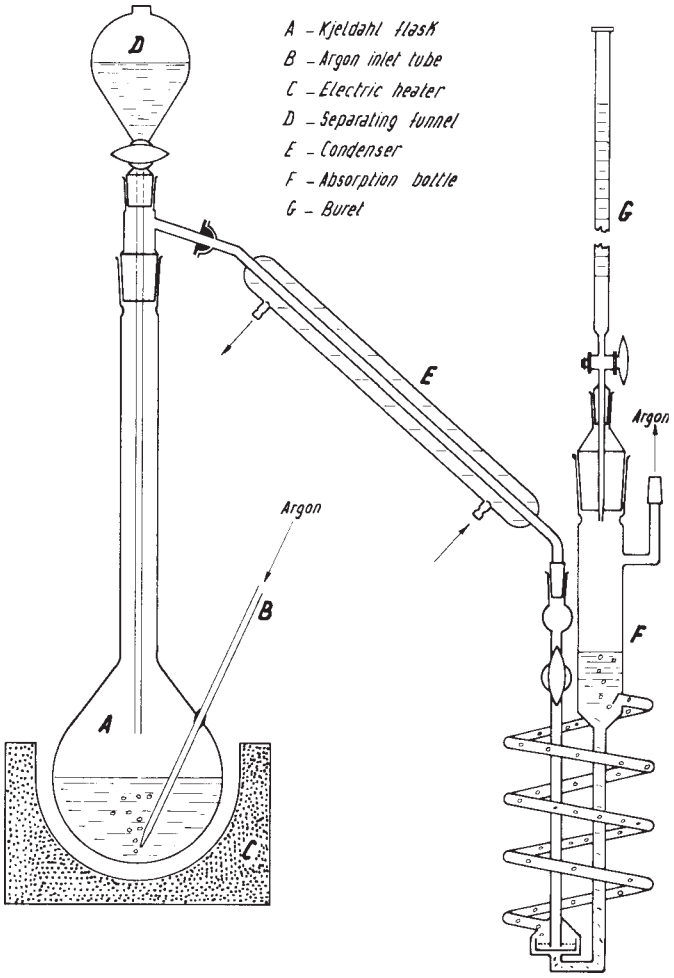


FIG. 4 Apparatus for Determination of Nitrogen Content

11.8.3.4 *Sulfuric Acid, Standard* (0.05 N)—Prepare and standardize 0.05 *N* sulfuric acid (H_2SO_4).

11.8.3.5 *Sodium Hydroxide Solution, Standard* (0.05 N)—Prepare and standardize a 0.05 *N* solution of sodium hydroxide (NaOH).

11.8.4 *Procedure*—Transfer 5 g of the getter alloy to the Kjeldahl flask. Add 50 cm^3 of the H_2SO_4-H_3PO_4 mixture. Heat until the sample is dissolved and then evaporate until white fumes are distinctly visible. Without cooling, rinse the neck of the flask with 5 cm^3 of concentrated H_2SO_4 (sp gr 1.82) by means of a pipet. Digest for about 2 h, regulating the heat so that the solution boils gently and there is a slight amount of fumes escaping at the outlet of the flask. Cool somewhat and add 200 cm^3 of water. Stir and heat gently to dissolve salts as completely as possible (avoid evaporation of much water). Rinse the separatory funnel, the condenser, and the absorption bottle with water and connect to the Kjeldahl flask. Place about 15 cm^3 of water in the absorption bottle and 100 cm^3 of the NaOH solution (333 g/L) in the separatory funnel. Pass a gentle stream of argon through the apparatus and add to the absorption bottle, by means of a buret, 25 cm^3 of the 0.05 *N* H_2SO_4 and a few drops of the indicator solution. Drain cautiously, drop by drop, the NaOH solution into the acid, rinse the separatory funnel, with a few cubic centimeters of water

and heat. Distill, as rapidly as possible, until about 75 cm^3 of distillate is collected in the absorption bottle. Titrate with the 0.05 *N* NaOH solution until the color changes from wine-red to green.

11.8.5 *Calculation*—The percent of nitrogen in the sample is given as follows:

$$\text{Nitrogen, percent} = [(V_1N_1 - V_2N_2)\ 0.7/W] \times 100$$

where:

V_1 = cm^3 of H_2SO_4 added to absorption bottle,
N_1 = normality of the H_2SO_4,
V_2 = cm^3 of NaOH solution required for titration,
N_2 = normality of the NaOH solution, and
W = milligrams of sample used.

11.8.6 The accuracy is ±10 %.

11.9 *Report*:

11.9.1 The getter gas content report includes:

11.9.1.1 Manufacturer's standard getter nomenclature,

11.9.1.2 Getter lot number, and

11.9.1.3 The measured gas content: total gas content as nitrogen (11.4) or preflash gas content as nitrogen (11.5) or both, total hydrogen content as hydrogen if applicable (11.7), and total nitrogen content as nitrogen if applicable (11.8).

11.9.2 Report the standard deviation and the number of samples.

11.10 The precision is ±10 %.

12. Determination of Carbon Monoxide Sorption Characteristics

12.1 *Summary of Method*—The sorption efficiency of a getter is rated by two characteristics: gettering rate and sorption capacity. These are determined dynamically from the instantaneous values of the gas mass throughput into the flashed barium film. Carbon monoxide is used as the test gas. The CO being sorbed must flow through a known conductance.

12.1.1 The measurement of the sorption characteristics, in particular the gettering rate, is complicated and difficult to execute. In fact, the measurements are influenced by the position of both the gas inlet and the P_g pressure gage with respect to the barium film, other conductances within the system and the presence of gases other than CO. There are three broad areas of application of CO sorption measurements: comparison between getter types, getter performance in a specific electron device, and basic studies of getter film properties. In general one is more interested in the reproducibility of the CO sorption capacity in the first two cases. As a result, there can be variations in the apparatus used since relative values of terminal gettering rates are adequate to define the sorption end point and also yield some information as to the surface area-porosity properties of the film. However, it is recommended that the form and dimensions of the detailed apparatus be adhered to in all cases.

12.1.2 The sorption characteristics are adversely affected by the gases emitted by the apparatus (leak-up rate). The recommended vacuum levels should therefore be carefully maintained.

12.1.3 The position of the getter (its distance from the yoke reference line *YRL*, for TV bulbs) and flashing conditions must be controlled to a high accuracy to assure the maximum reproducibility of results. This is because the sorption characteristics are influenced by the surface area of the film which depends upon the getter position, and by the porosity which depends upon both the flashing conditions and inherent getter characteristics.

12.1.4 The sorption tests may be carried out at constant throughput, Q, constant manifold pressure, P_m, or at constant pressure above the getter, P_g. Constant P_m is practically similar to constant Q. However, the terminal portion of the sorption curve obtained at constant Q will be slightly different.

12.1.4.1 Tests that serve as a comparison basis between different getter types or that evaluate getter behavior in a given electron device can be performed at constant P_m since the experiment is simpler to run and the values obtained are practically near the true values.

12.1.4.2 When basis studies of sorption mechanisms or calculations of activation energies are required the experiment should be performed at constant P_g as the diffusion of CO into the interior of the film is the rate limiting factor and diffusion depends on pressure.

12.1.5 Advantage can be taken of the stringent vacuum requirements of the sorption apparatus to measure the relative quantities of unpumpable gases released or produced as a consequence of getter flashing by measuring the pressure during and after flashing. This measurement of nonsorbable gases is extremely critical and the data obtained is unreliable unless low-ionizing electron currents are used. Otherwise the data refers to the pumping action of the pressure gage.

12.2 *Significance*:

12.2.1 The CO sorption capacity of a getter gives a measure of the amount of gas which a getter can sorb.

12.2.2 The short-term CO sorption capacity depends more on the film area and porosity than on the amount of barium present since the diffusion of CO into the bulk of the getter is the rate limiting reaction. In order to obtain reproducible results, the test apparatus and procedures must be tightly controlled.

12.2.3 The gettering rate gives a measure of the ability to maintain a desired vacuum against the outgassing of system components.

12.3 *Apparatus*:

12.3.1 Typical vacuum systems for receiving-tube and picture-tube getters are shown schematically in Fig. 5. Fig. 6 details the bulb and pressure-gage layout for receiving-tube getters and Fig. 7, for picture-tube getters. The inlet CO is directed at the barium film. The P_g pressure gage is positioned to measure the CO pressure above the barium film after the CO has collided with the film surface. The pressure gages are located so that the flowing CO is not directed into the gages and the conductance of their connecting tubulation must be large enough so that pressure drops caused by gage pumping is negligible.

12.3.2 The system must be capable of reaching an ultimate pressure of less than 1.10^{-8} Torr. The leak-up rate should be less than 1.10^{-6} mtorr·litre/s. An oil diffusion pump is recommended but back migration of oil vapors must be prevented by using suitable traps. Pumps which have selective pumping action toward different gases are not recommended.

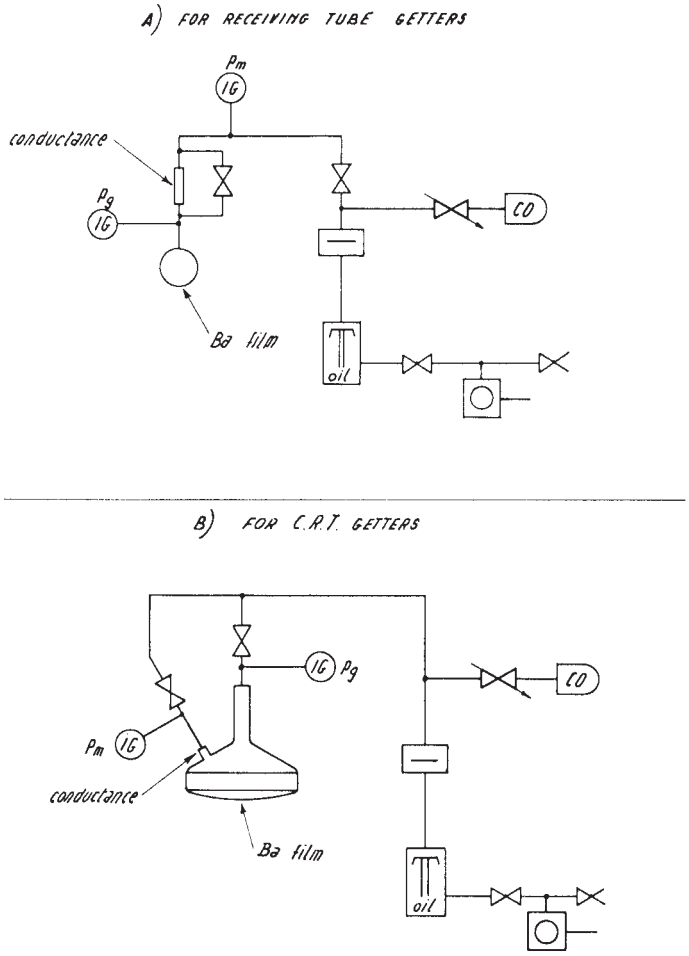
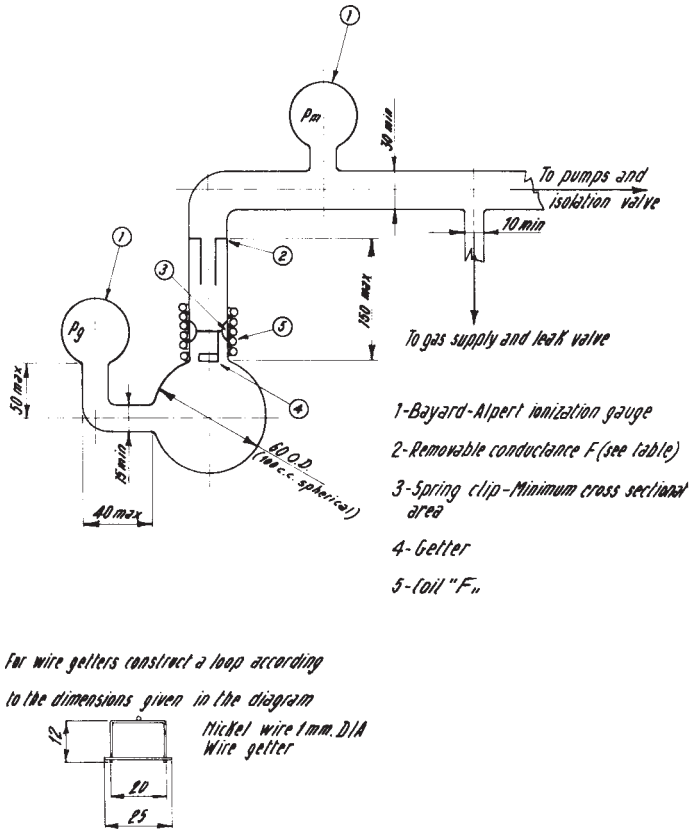


FIG. 5 Schematic of Sorption Measurement Apparatus



NOTE 1—All dimensions are in millimetres.

FIG. 6 Apparatus for Determination of CO Sorption Characteristics: Receiving Tube Getters

12.3.3 The test bulb must be connected to the manifold by means of bakeable hermetic seals. The bulbs and coils to be used are listed in Table 2. The recommended getter mountings are:

Receiving tube loop	Directly onto spring mount
Receiving tube ring	Directly onto spring mount
Picture tube getters	The last grid of the appropriate gun as normally used in the TV bulb in which the getter is tested or spring mount

12.3.4 The pressure gages for both P_m and P_g should be of the Bayert-Alpert type (BAG) and used with a maximum electron ionizing current of 100 μA. A low-temperature emitter is preferred. One of the gages must be calibrated against an absolute McLeod gage using CO as the gas. (A partial pressure analyzer can be used to measure P_s.) Larger ionizing currents can be used providing that there is no evidence of pumping by the pressure gage.

12.3.5 The carbon monoxide gas shall be the purest available (99.8 % CO min). The purity of the gas is very important since slowly sorbable impurities will build up in the getter chamber causing the pressure above the getter to increase and hence yield apparently poor gettering characteristics.

12.3.6 The getter position within the test bulb is given in Table 3.

12.3.7 The known conductance, *F*, is chosen in relation to the type of getter and measuring system. Its value, when measuring at P_m constant, must ensure that P_g is accurately measurable and therefore greater than the residual pressure of the system. Conductance values for suggested values of P_m constant are given in Table 5. In the case of testing at P_g constant the conductance values must ensure that P_m is not so high at the beginning of the test that molecular flow no longer applies.

12.3.8 *Calculation of Molecular Flow Conductance*:

12.3.8.1 The conductances to consider are: a tube of length, *l*, and inner radius, *a*, and a circular hole of radius *a* in material of thickness, *T*.

12.3.8.2 The conductance in litres per second is given as follows:

$$F = (3.638\pi a^2 K)(T/M)^{1/2}$$

where:

T = absolute temperature, K,
M = molecular weight of the gas, g,
K = Clausing's correction factor (a function of l/a), and
a = radius, cm.

A table of *K* values for various *l/a* values is given in Table 6.

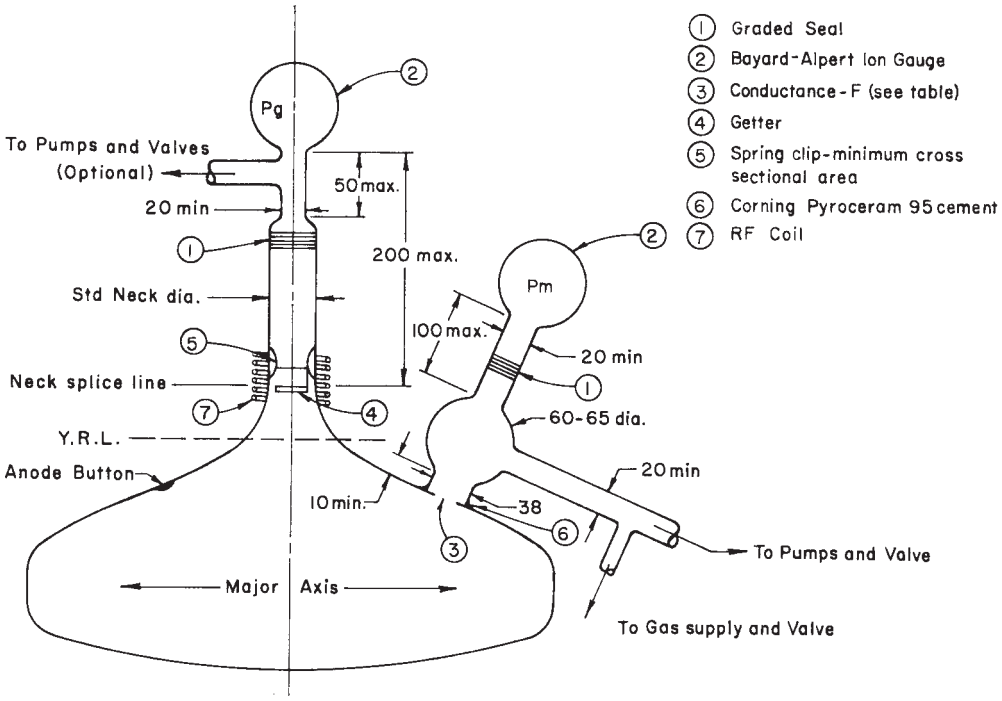
12.4 *Procedure*:

12.4.1 Observe the clean handling techniques of 5.5.1.

12.4.2 Weigh the getter with its integral support.

12.4.3 Attach the test getter to its appropriate cleaned mount.

12.4.4 Secure the mounted getter in the test apparatus.



NOTE 1—All dimensions are in millimetres.

FIG. 7 Sorption Apparatus Layout for TV Getters

TABLE 5 Conductance for Suggested P_m Values

Test Bulb	Suggested P_m, Torr	Conductance, litres/s
100 cm^3 spherical	$1.0\ 10^{-4}$	0.2
12 in., 110°, 20-mm neck	$7.0\ 10^{-5}$	3
19 in., 114°, 28.5-mm neck	$7.0\ 10^{-5}$	7
23 in., 92°, 36.5-mm neck	$1.2\ 10^{-4}$	9

TABLE 6 K values for 1/*a* (see 12.3.8.2)

1/*a*	*K*	1/*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	...	8*a*/31

12.4.5 Close the system by hermetic sealing and pump while the system is baked at 300 ± 15°C (bulb or face plate temperature) over night or longer so that a pressure of less than 1.10^{-8} Torr is reached.

12.4.6 Degas the ionization gages.

12.4.7 Isolate the pumping system and measure the leak-up rate using the P_g ionization gage. If less than 1.10^{-6} mtorr·litre/s, repump the system to less than 1.10^{-8} Torr.

12.4.8 During each test calibrate the second gage, with respect to the one previously calibrated by the McLeod by allowing CO into the system under pumping. Each decade within the measurement range must contain at least one calibration point.

12.4.9 Flash the getter with start and total times such as to give the desired barium yield. Flashing is to be performed as follows:

12.4.9.1 The bulb must be at ambient temperature and only the P_g BAG must be functioning at the least possible emission current (10 µA). The 10-µA ionizing current is desirable since it permits measuring the peak preflash pressure on a relative basis and also minimizes gage pumping in determining non-sorbable gases produced as a result of the getter flash. Larger ionizing currents can be used providing that there is no indication of ionization gage pumping.

12.4.9.2 Isolate the system from the pump.

12.4.9.3 Flash the getter. Measure the pressure by the P_g BAG and record the maximum value during flash. This value is a comparative value of the gas content only and is not absolute without gage calibration.

12.4.9.4 After ¼ h remeasure the pressure with the P_g BAG. This pressure is a measure of the nonsorbable gases released.

12.4.10 Open the pump isolation valve and repump until the pressure is less than 5.10^{-8} Torr.

12.4.11 Ensure that the two BAGs are switched on at 100 µA. Admit CO until the required P_g or P_m is reached in the shortest possible time. Immediately record the other pressure. Larger ionizing currents can be used providing that there is no indication of pumping by the ionization gages.

12.4.12 Continue the test maintaining the pressure (P_g or P_m) at the initial value, recording periodically the other pressure (P_m or P_g) and the elapsed time or record it continuously. The test is terminated when the terminal gettering rate is reached (1 L/s for TV getters or 0.1 L/s for receiving-tube getters).

12.4.13 Let the system down to air.

12.4.14 Remove the getter and weigh.

12.5 *Calculations*:

12.5.1 *Gettering Rate*—The instantaneous value of the gettering rate, *G*, in litres per second, is given as follows:

$$G = F\,(P_m - P_g)/P_g$$

where:

F = known (calculated) conductance, Ls/s,
P_m = manifold (inlet) pressure of CO, mtorr, and
P_g = pressure above the getter film, mtorr.

If: $P_m \geq P_g$ then: $G \approx FP_m/P_g$.

12.5.2 *The sorption capacity, C*, in micrometre-litres is given by the following equation:

$$C = F \int (P_m - P_g)\mathrm{d}t$$

where:

t = time, s.

The $\int \mathrm{d}t$ is evaluated over the total sorption time.

12.5.2.1 *C* can evaluated manually, graphically, or by computer.

12.5.2.2 The sorption capacity can be approximated by dividing the total sorption time into a number of smaller time intervals, t_i. Then:

$$C_i = F\,(p_m - p_g)t_i$$

where:

$(p_m - p_g)$ = average pressure difference, mtorr, in the interval t_i. (The final pressure difference in the interval t_i can be used instead and causes an error of less than 5 % in the value of the getter sorption capacity.)

and

$$C = \Sigma_i C_i \text{ mtorr·litres}$$

If $P_m \geq P_g$ then: The sorption capacity in millitorr-litres can be approximated by the following equation:

$$C \approx FP_m t$$

12.5.3 *The Specific Sorption Capacity* is given as follows:

$$C_{sp} = C/M_y$$

where M_y = milligrams of evaporated barium.

12.5.4 *Carbon Monoxide Sorptive Curve*—Plot the logarithmic gettering rate versus the quantity of CO sorbed. The X-axis represents the amount of CO sorbed in millitorr-litres and ranges from zero to the theoretical capacity. The Y-axis represents the logarithm of the gettering rate in litres per second and must cover at least four orders of magnitude. The graph should show the following information:

12.5.4.1 Manufacturer's standard getter nomenclature and lot number (if applicable),
12.5.4.2 Average yield (mg),
12.5.4.3 Start and total time (s),
12.5.4.4 Flashing frequency (kHz),
12.5.4.5 Conductance value used,
12.5.4.6 Whether P_m or P_g constant and its value,
12.5.4.7 Type of test bulb and coil,
12.5.4.8 End point gettering rate,
12.5.4.9 CO sorption capacity,
12.5.4.10 Specific CO sorption capacity, and
12.5.4.11 Gas emission on flashing.

12.6 *Report*—The report will consist of:

12.6.1 A typical sorption curve and

12.6.2 The average specific sorption capacity, its standard deviation, and the number of samples used.

12.7 The precision is ±10 %.

13. Keywords

13.1 barium content; barium yield; getter materials; getter sorption capacity

APPENDIX

(Nonmandatory Information)

X1. CURRENT AMERICAN AND EUROPEAN GETTER SIZES

X1.1 Data for the current American and European getters are given in Table X1.1, Table X1.2, and Table X1.3.

TABLE X1.2 Ring Getters for Receiving Tubes

Channel, Outside Diameter, mm	Total Ba in Fill (ma)	
	Exothermic	Endothermic
14	4.0–11.5	...
12/13	3.5–25	7.5
11	3.0–11	6.0
10	3.0–7	...
9.5	2.5–7.5	5.0
7	4.0–5	...
5	1.0–2.5	...
4	0.5–1	...

TABLE X1.1 Ring Getters for Picture Tubes

Channel, Outside Diameter, mm	Total Barium in Fill (mg)		Typical Use
	Exothermic	Endothermic	
36	...	150	70° color
25 gun	85–170	130–150	90° color
25 ant	140–280	...	90° color
22/23	45–110	100	70–90° black and white
18/19	30–85	90–150	110–114° black and white
12/13	12–39	...	narrow neck black and white

TABLE X1.3 Getter Wire Data

Maximum Cross Section, mm	Barium Fill, mg/cm of length[A]		
	Exothermic	Endothermic	Pure Barium
0.50	1.0–3.1	2.0	2.5
0.60	1.7	4.0	4.0
0.75	1.0–3.1	2.0	...
0.85	1.5	3.0	...
1.17	3.5	6.0	...
1.20	8.7	...	...

[A]The active wire length of loop-type getters may vary continuously between 2 and 20 mm. The barium fill is calculated from the wire data.