Designation: E 834 – 81 (Reapproved 1998)

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Determining Vacuum Chamber Gaseous Environment Using a Cold Finger[1]

This standard is issued under the fixed designation E 834; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a technique for collecting samples of materials that are part of the residual gas environment of an evacuated vacuum chamber. The practice uses a device designated as a "cold finger" that is placed within the environment to be sampled and is cooled so that constituents of the environment are retained on the cold-finger surface.

1.2 The practice covers a method for obtaining a sample from the cold finger and determining the weight of the material removed from the cold finger.

1.3 The practice contains recommendations as to ways in which the sample may be analyzed to identify the constituents that comprise the sample.

1.4 By determining the species that constitute the sample, the practice may be used to assist in defining the source of the constituents and whether the sample is generally representative of samples similarly obtained from the vacuum chamber itself.

1.5 This practice covers alternative approaches and usages to which the practice can be put.

1.6 The degree of molecular flux anisotropy significantly affects the assurance with which one can attribute characteristics determined by this procedure to the vacuum chamber environment in general.

1.7 The temperature of the cold finger significantly affects the quantity and species of materials collected.

1.8 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[2]

3. Terminology

3.1 *pretest cold finger sample residue mass, M_i*—the mass of material collected from the cold finger during the pretest operation and as measured by the techniques specified in Section 9. The mass is based on a sample volume of 50 mL.

3.2 *posttest stock sample residue mass, M_f*—the mass of residue in a sample collected from the cold finger during the posttest operation and as measured by the technique specified in Section 9. The mass is based on a sample volume of 50 mL.

3.3 *pretest stock sample residue mass, S_i*—the mass of residue in a sample of the solvent (used to obtain the pretest cold finger sample) as measured by the technique specified in Section 9. The mass is based on a sample volume of 50 mL.

3.4 *posttest stock sample residue mass, S_f*—the mass of residue in a sample of the solvent (used to obtain the posttest cold finger sample) as measured by the technique specified in Section 9. The mass is based on a sample volume of 50 mL.

3.5 *cold finger*—the device that is used in collecting the sample of the residual gases in an evacuated vacuum chamber (see Fig. 1).

3.6 *CFR*—the residue collected by the cold finger during the vacuum exposure given in milligrams.

4. Summary of Practice

4.1 The cold-finger technique provides a method for characterizing the ambiance in a vacuum chamber when the chamber is being operated with or without a test item.

4.2 In use, the cold finger is installed in the vacuum chamber in such a location as to be exposed to fluxes representative of those in the general ambiance. (Chamber conditions that will exist under vacuum conditions must be considered so as to assess the effects of molecular flux anisotropy.)

4.3 The cold finger is cleaned before the vacuum exposure and a sample of any residue on the surface is obtained. The pretest cleaning and sampling procedure consists of (*a*) heating the cold finger and scrubbing it with a solution of laboratory detergent and water; (*b*) rinsing the cold finger with demineralized or distilled water; (*c*) rinsing the cold finger with 1,1,1-trichloroethane and ethanol mixed 75 + 25 as the solvent; (*d*) obtaining a sample of any residue contained in a second rinse with solvent; and (*e*) obtaining a sample of the solvent.

4.4 The vacuum chamber is then sealed and evacuated; after reaching a pressure of less than 1 mPa (8×10^{-6} torr), a coolant is flowed through the cold finger so that materials in the ambient environment can adhere to the surface. Generally,

[1] This practice is under the jurisdiction of ASTM Committee E-21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.05 on Contamination.

Current edition approved Sept. 25, 1981. Published December 1981.

[2] *Annual Book of ASTM Standards*, Vol 14.02.

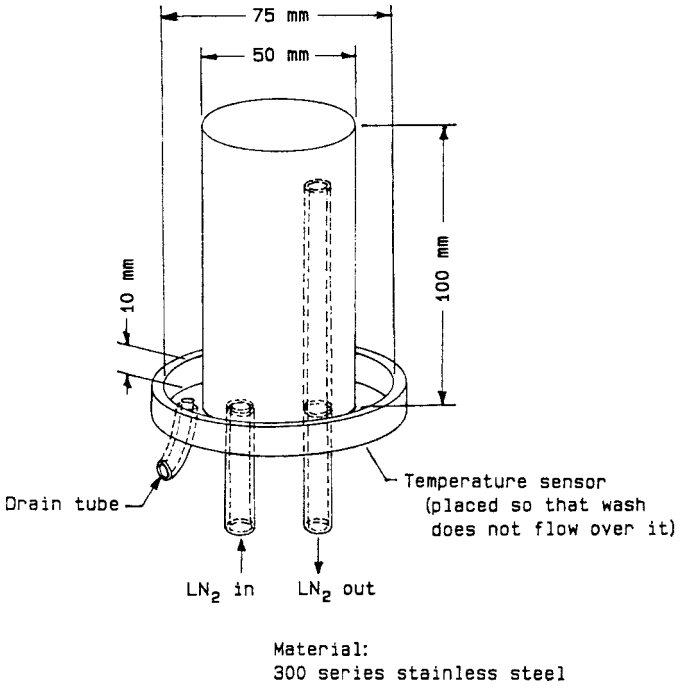

FIG. 1 Typical Cold Finger Assembly

liquid nitrogen is used as the coolant. Other coolants may be used provided that the coolant temperature is controlled and reported. This coolant flow is continued until the chamber pressure rises to greater than 80 kPa (600 torr) as the chamber is being returned to room ambient conditions using dry gaseous nitrogen. **Caution:** Too rapid a repressurization may dislodge some of the condensate.

4.5 As soon as possible after the chamber door is opened, the solvent is poured over the cold finger and a sample containing any residue from the cold finger is collected. A second sample of the solvent is obtained if the solvent is taken from a container different than that used under 4.3.

4.6 Both the pretest and posttest samples are placed in previously cleaned and weighed evaporating dishes. The dishes containing the samples are placed on a steam bath and the solvent is evaporated. The dishes containing the residue are then weighed using an analytical balance. The samples of the solvent are similarly handled and any residue weighed. The differences of mass between the pretest residue and posttest residue is then determined (corrected if necessary for any significant residue found in the solvent); this difference in mass is taken as the residue collected by the cold finger during its exposure to the vacuum environment, CFR.

4.7 Analytical procedures such as infrared spectroscopy or gas chromatography-mass spectrometry may be used to identify those species that constitute the residue.

5. Significance and Use

5.1 When applied in the case in which there is no test item in the vacuum chamber (such as during bake-out operations), this procedure may be used to evaluate the performance of the vacuum chamber in relation to other data from the same or other chambers given that critical parameters (for example, length of exposure, temperature of the chamber and cold finger, anisotropy, and so forth) can be related.

5.2 The procedure can be used to evaluate the effects of materials found in the residue on items placed in the vacuum chamber.

5.3 The procedure can be used to describe the effect of a prior test on the residual gases within a vacuum chamber.

5.4 By selecting the time at which the coolant is introduced into the cold finger, the environment present during a selected portion of a test can be characterized. This can be used to determine the relative efficacy of certain vacuum chamber procedures such as bake-out.

5.5 The procedure may be used to define the outgassed products of a test item that condense on the cold finger.

5.6 The procedure may be used in defining the relative cleanliness of a vacuum chamber.

5.7 In applying the results of the procedure to the vacuum chamber in general, consideration must be given to the anisotropy of the molecular fluxes within the chamber.

5.8 The procedure is sensitive to both the partial pressures of the gases that form the condensibles and the time of exposure of the cold finger at coolant temperatures.

5.9 The procedure is sensitive to any losses of sample that may occur during the various transfer operations and during that procedure wherein the solvent is evaporated by heating it on a steam bath.

NOTE 1—Reactions between solvent and condensate can occur and would affect the analysis.

6. Apparatus

6.1 The apparatus used in this procedure is termed a cold finger. Fig. 1 is a drawing of the cold finger. The cold finger consists of a stainless steel cylinder approximately 50 mm (2.0 in.) in diameter and 100 mm (4.0 in.) high. The base of the cylinder is extended to form a lip or trap annulus approximately 10 mm (½ in.) high with a diameter of 75 mm (3 in.) so that fluid poured over the top of the cylinder and running down the sides can be captured. A small drain is provided in this lip and the fluid can drain through this aperture into a receptacle. Two tubes enter the cold finger through the base, one providing the inlet and the other the outlet for the coolant. Temperatures shall be monitored. The coolant recommended in this practice is liquid nitrogen. The apparatus should be thoroughly cleaned after the manufacture.

6.2 Containers must not react with the solvents. Glass, austenitic stainless steels, or PTFE generally are acceptable.

7. Reagents

7.1 Reagent grade 1,1,1-trichloroethane and ethanol mixed 3 + 1 is the solvent used for obtaining the sample from the cold finger and as the final rinse material in the cleaning procedures for the various equipment that will come in contact with the sample during the execution of this practice.

8. Precautions

8.1 Equipment other than the cold finger that will come in contact with samples should be cleaned in accordance with the annex to this practice.

8.2 The cold finger should never be touched with bare hands after cleaning.

9. Procedure

9.1 *Cleaning the Cold Finger*—The cold finger should be thoroughly cleaned when installed and after each test to ensure that contamination is not carried from test to test. The cleaning procedure should be as follows:

9.1.1 Heat the cold finger with an electric torch or flexible heater to approximately 60°C.

9.1.2 Scrub the cold finger with a solution of laboratory detergent[3] and hot distilled water using an extracted, lint-free wiping pad. It should be cleaned on all surfaces, plus approximately 50 mm (2 in.) of the coolant lines where they enter the cold finger.

9.1.3 Rinse the cold finger with hot, clean, distilled water. Particular attention should be given to the corners of the annulus and its drain hole as well as the welding relief groove on the top.

9.1.4 Flood rinse all washed surfaces with solvent. The electric torch may be used to assist the drying action.

9.1.5 Discard all used wash and rinse fluids.

9.1.6 Cover the cold finger with a piece of cleaned aluminum foil or lint-free cloth if the wash sample is not to be taken at once.

9.2 *Taking the Pretest Cold Finger Sample*:

9.2.1 Pour approximately 100 mL of solvent over the cold finger. (Do not splash alcohol on the chamber shroud.) Pour at such a rate that the trap annulus is filled to overflowing. Catch this fluid in a basin or similar container and discard it.

9.2.2 Pour 50 mL of the solvent over the cold finger. Do not overflow the trap annulus. Catch the solvent directly with a *clean* sample bottle. Label this bottle *Pretest Sample.*

9.2.3 Pour 50 mL of solvent (Note 2) into a *clean* sample bottle directly from the same container used to pour it over the cold finger. Label this bottle *Pretest Stock.*

NOTE 2—If experience indicates the solvent to yield consistently less than 0.2 mg of residue, the steps indicated in 9.2.3 and 9.4.2 need be done only when a new container of solvent is used.

9.3 *Chamber Operations*:

9.3.1 If any protective cover has been placed over the cold finger, it should be removed immediately before the chamber door is closed.

9.3.2 Coolant should be admitted to the cold finger when the chamber pressure decreases below 1 mPa (8×10^{-6} torr), and flow should be continued to maintain the cold finger at a stable temperature until the chamber return to atmosphere is underway. The temperature of the cold finger should be monitored.

9.3.3 The coolant flow should be terminated when the chamber pressure rises above 80 kPa (600 torr) during the return to room ambient conditions using gaseous nitrogen. The temperature of the cold finger should be kept above the dew point of water in the ambience during the return to atmosphere and after the chamber door is opened.

9.4 *Taking the Posttest Cold Finger Sample*:

9.4.1 As soon as possible after the chamber is open, pour 50 mL of solvent over the cold finger. Catch the solvent directly with a *clean* sample bottle. Label this bottle *Posttest Sample.*

9.4.2 Pour 50 mL of solvent (Note 2) from the same container *directly* into a *clean* sample bottle. Label this bottle *Posttest Stock.* (This step may be omitted if the solvent is taken from the same container as that in 9.3.2.)

9.5 *Evaporating and Weighing*—This section applies to pretest and posttest cold finger and stock samples.

9.5.1 Weigh a cleaned porcelain evaporating dish (about 75 mm in diameter) using an analytical balance having accuracy and a precision of at least 0.1 mg.

9.5.2 Place the entire sample in the evaporating dish.

9.5.3 Place the evaporating dish containing the sample in a steam bath[4] and heat the dish until the solvent has been evaporated.

9.5.4 Weigh the evaporating dish containing any residue from the sample using a balance as in 9.5.1.

9.5.5 **Caution:** The evaporating dish should not be handled with bare hands so that skin oils or other contaminants are not transferred to the dish.

9.5.6 **Caution:** Weighing should be done after the evaporating dishes have reached room temperature.

9.6 *Other Analysis*—The residue that remains in the evaporating dish may be subjected to chemical analysis such as infrared spectroscopy or gas chromatography-mass spectrometry so as to identify those species that constitute it; a relative quantitazation among species is often helpful.

10. Calculation

10.1 Calculate CFR as follows:

10.1.1 Determine the mass of the residue in stock samples by subtracting the mass of the empty evaporating dish from the mass of the evaporating dish after the stock sample has been evaporated. Designate the pretest stock residue as S_i and the posttest residue as S_f, both expressed in milligrams. If either S_i or S_f are found to be greater than 0.2 mg for a 50-mL sample, their effect should be considered; if not, they may be neglected from the calculations.

10.1.2 Determine the mass of the residue in the cold finger sample by subtracting the mass of the empty evaporating dish from the mass of the dish after the cold finger sample has been evaporated. Designate the pretest cold finger sample residue as M_i and the posttest cold finger sample residue as M_f, both expressed in milligrams.

10.1.3 The mass of the residue collected by the cold finger during its exposure to vacuum and remaining after processing to this part, CFR, is expressed in milligrams:

$$CFR = (M_f - S_f) - (M_i - S_i) \quad (1)$$

11. Report

11.1 The report of the results of the cold finger procedure should contain the following information:

11.1.1 The name of the organization conducting the procedure.

11.1.2 The date of the weighing.

11.1.3 The designation and short description of the facility in which the cold finger sample was obtained.

[3] Alconox has been found to be a satisfactory detergent.

[4] Fisher Scientific electrically heated water bath, Catalog No. 15496, is a suggested type.

11.1.4 A short description of the purpose of the vacuum exposure; one sentence will generally suffice.

11.1.5 The dates and times over which the sample was obtained and the total number of hours the cold finger was being cooled.

11.1.6 The temperature of the cold finger during the vacuum exposure; the measurement technique and estimated accuracy.

11.1.7 The masses of the stock sample residues, S_i and S_f expressed in milligrams; if both S_i and S_f are equal or less than 0.2 mg, this should be noted and specific masses need not be provided.

11.1.8 The masses of the cold-finger sample residues, M_i and M_f, expressed in milligrams.

11.1.9 The mass of the residue collected during the vacuum exposure, CFR.

11.1.10 The results of any chemical analysis that may have been conducted to characterize the residue further. The analytical technique and the particular instrument used should be identified.

11.1.11 Any other pertinent information that the supplier considers relevant.

12. Precision and Bias

12.1 Neither the precision nor the bias for this method has been determined.

12.2 The 50-mL samples should have a precision of ±2 mL.

12.3 Weighings should be made to the nearest 0.1 mg.

12.4 Results should be expressed to the nearest 1 mg.

ANNEX

(Mandatory Information)

A1. CLEANING

A1.1 Equipment Required

A1.1.1 *Noncontaminating Plastic Wash Tray*.

A1.1.2 *Noncontaminating Plastic Drain Tray.*

A1.1.3 *Wire Drying Rack.*

A1.1.4 *Laboratory Detergent*[3].

A1.1.5 *Hot Tap Water*.

A1.1.6 *1,1,1-Trichloroethane and Ethanol* (mixed 3 + 1).

A1.1.7 *Pressurizable Container* for the solvent.

A1.1.8 *Cotton Gloves*.

A1.1.9 *Plastic Gloves* (not vinyl or any extractable plasticized type).

A1.1.10 *Nylon Bottle Brush*.

A1.1.11 *Distilled Water.*

A1.1.12 *Extracted Lint-Free Cloths*.

A1.2 Procedure

A1.2.1 Wash items A1.1.1, A1.1.2, A1.1.3, and A1.1.10 in a hot solution of laboratory detergent and water and rinse them in hot water.

A1.2.2 Prepare a hot solution of laboratory detergent and water in the plastic wash tray.

A1.2.3 Soak the bottles (or evaporating dishes) in this solution for 10 min.

A1.2.4 Scrub the bottles with the nylon brush; wash the evaporating dishes using the lint-free cloths; use cotton gloves inside the plastic ones.

A1.2.5 Rinse with hot tap water four times.

A1.2.6 Rinse with distilled water three times.

A1.2.7 Rinse with solvent from the pressurized vessel three times.

A1.2.8 Shake excess from bottle (evaporating dish) and store, mouth down, on the wire drying rack.

A1.2.9 Place glass stoppers in a plastic bag and clean several at one time repeating steps A1.2.3-A1.2.8 for the stoppers.

A1.2.10 Place stoppers on drying rack.

A1.2.11 When stoppers and bottles are dry, place a stopper in each bottle and seal with a strip of clear tape over the stopper, making sure that the tape adhesive does not adhere to the lip of the bottle. (Place the evaporating dishes in previously similarly cleaned aluminum foil or in small cabinets until needed; if they are not used immediately, rinse them with solvent and allow them to dry before using.)