EXHIBIT 150
PART 12

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Standard Practice for Packed Column Gas Chromatography[1]

This standard is issued under the fixed designation E 260; 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 is intended to serve as a general guide to the application of gas chromatography (GC) with packed columns for the separation and analysis of vaporizable or gaseous organic and inorganic mixtures and as a reference for the writing and reporting of GC methods.

NOTE 1—This practice excludes any form of gas chromatography associated with open tubular (capillary) columns.

1.2 *This standard does not purport to address all the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7 and 9.1.3.

2. Referenced Documents

2.1 *ASTM Standards:*

E 355 Practice for Gas Chromatography Terms and Relationships[2]
E 516 Practice for Testing Thermal Conductivity Detectors Used in Gas Chromatography[2]
E 594 Practice for Testing Flame Ionization Detectors Used in Gas Chromatography[2]
E 697 Practice for Use of Electron Capture Detectors in Gas Chromatography[2]
E 840 Practice for Using Flame Photometric Detectors in Gas Chromatography[2]
E 1140 Practice for Testing Nitrogen/Phosphorus Thermionic Ionization Detectors for Use in Gas Chromatography[2]

2.2 *CGA Publications:*

CGA P-1 Safe Handling of Compressed Gases in Containers[3]
CGA G-5.4 Standard for Hydrogen Piping Systems at Consumer Locations[3]
CGA P-9 The Inert Gases: Argon, Nitrogen and Helium[3]
CGA V-7 Standard Method of Determining Cylinder Valve Outlet Connections for Industrial Gas Mixtures[3]
CGA P-12 Safe Handling of Cryogenic Liquids[3]
HB-3 Handbook of Compressed Gases[3]

3. Terminology

3.1 Terms and relations are defined in Practice E 355 and references therein.

4. Summary of Practice

4.1 A block diagram of the basic apparatus needed for a gas chromatographic system is as shown in Fig. 1. An inert, pressure or flow-controlled carrier gas flowing at a measured rate passes to the injection port or gas sample valve. A sample is introduced into the injection port, where it is vaporized, or if gaseous, into a gas sample valve, and then swept into and through the column by the carrier gas. Passage through the column separates the sample into its components. The effluent from the column passes to a detector where the response of sample components is measured as they emerge from the column. The detector electrical output is relative to the concentration of each resolved component and is transmitted to a recorder, or electronic data processing system, or both, to produce a record of the separation, or chromatogram, from which detailed analysis can be obtained. The detector effluent must be vented to a hood if the effluent contains toxic substances.

4.2 Gas chromatography is essentially a physical separation technique. The separation is obtained when the sample mixture in the vapor phase passes through a column containing a stationary phase possessing special adsorptive properties. The degree of separation depends upon the differences in the distribution of volatile compounds, organic or inorganic, between a gaseous mobile phase and a selected stationary phase that is contained in a tube or GC column. In gas-liquid chromatography (GLC), the stationary phase is a nonvolatile liquid or gum coated as a thin film on a finely-divided, inert support of a relatively large surface area, and the distribution is based on partition. The liquid phase should not react with, and should have different partition coefficients for, the various components in the sample. In gas-solid chromatography (GSC), the stationary phase is a finely divided solid adsorbent (see 4.4).

4.2.1 After separation in the analytical column, the components are detected, and the detector signal is related to the concentration of the volatile components. Tentative identifications can be made by comparison with the retention times of known standards under the same conditions, either on a single column or preferably by injecting the sample onto two columns of different selectivity. Ancillary techniques, such as mass spectrometry or infrared spectrophotometry, are generally necessary for positive identification of components in samples.

4.2.2 Prior to performing a GC analysis, the following parameters must be considered:

4.2.2.1 Sample preparation.

4.2.2.2 Stationary phase and loading on support.

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[1] This practice is under the jurisdiction of ASTM Committee E-19 on Chromatography and is the direct responsibility of Subcommittee E19.03 on Methods and Specifications, Gas Chromatography.

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

[3] Available from Compressed Gas Association, Inc., 1725 Jefferson Davis Highway, Arlington, VA 22202-4100.

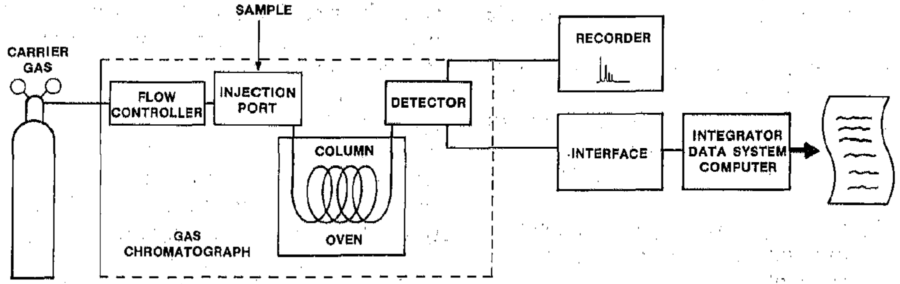

FIG. 1 Block Diagram of a Basic Gas Chromatographic System

4.2.2.3 Column material required.

4.2.2.4 Solid support and mesh size.

4.2.2.5 Column length and diameter.

4.2.2.6 Instrument and detector type that will be needed.

4.2.2.7 Injector, column oven, and detector temperatures required for analysis.

4.2.2.8 Injection techniques, such as flash volatilization, on-column technique, purge and trap, pyrolysis, etc.

4.2.2.9 Carrier gas and flow rate.

4.2.2.10 Data handling and presentation.

4.3 In gas-liquid chromatography, the degree of separation possible between any two compounds (solutes), is determined by the ratio of their partition coefficients and the separation efficiency. The partition coefficient, K, is the ratio of the solute concentration in the liquid phase to the solute concentration in the vapor phase at equilibrium conditions. The partition coefficient is affected by temperature and the chemical nature of the solute (sample) and solvent (stationary phase).

4.4 Another mechanism for separation is gas-solid chromatography. With this technique there is no liquid phase, only a porous polymer, molecular sieve, or solid adsorbent. Partition is accomplished by distribution between the gas phase and the solid phase.

4.5 After the sample is resolved into individual components by the chromatographic column, the concentration or mass flow of each component in the carrier gas can be measured by an appropriate detector which sends an electrical signal to a recording potentiometer or other readout device. The curve obtained by plotting detector response against time is referred to as a chromatogram. For flame ionization and thermal conductivity detectors, either the peak areas or the peak heights are proportional to the concentration of the components in the sample within the linear range of the detector system. However, response fractors are not necessarily the same for all compounds, and linearity of detector response may depend on operating conditions. (Testing of detector performance is discussed in ASTM Standard Practices for the appropriate detector, see 2.1).

4.6 Components in a mixture may be tentatively identified by retention time. Ideally, each substance has a unique retention time in the chromatogram for a specific set of operating conditions. However, caution is required because the GC separation may be incomplete and a single peak may represent more than one compound. This is especially true of unknown mixtures and complex mixtures because of the very large number of possible compounds in existence and the finite number of peaks that a chromatograph might resolve. Additional characterization data may be provided by ancillary techniques, such as spectrometry.

5. Significance and Use

5.1 This practice describes a procedure for packed-column gas chromatography. It provides general comments, recommended techniques, and precautions. A recommended form for reporting GC methods is given in Section 14.

6. Apparatus

6.1 *Carrier Gas System*—Common carrier gases are helium and nitrogen. Paragraph 7.6 provides more details on carrier gases. Means must be provided to measure and control the flow rate of the carrier gas. Any flow or pressure control and measurement combination may be used that will give an accurately known and reproducible flow rate over the desired range.

6.1.1 The main gas supply is regulated with a two-stage regulator which must have a stainless steel diaphragm. Rubber or plastic diaphragms permit oxygen or water to diffuse into the carrier gas. In addition, instruments will have a flow controller between the pressure regulator and column inlet to maintain a constant flow during temperature programming. Copper or stainless steel carrier gas lines, not plastic tubing, should be used to avoid diffusion of oxygen (air) into the carrier gas. When using the thermal conductivity detector, variations in the flow will change retention and response. The carrier gas line pressure must be higher than that required to maintain the column flow at the upper temperature limit for the flow controller to operate properly. A pressure of 40 to 60 psi is usually sufficient.

6.2 *Column Temperature Control*—Precise column temperature control is mandatory if reproducible analyses are to be obtained. Temperature control must be within 0.1°C if retention times are to be compared with another instrument.

6.2.1 *Air Bath*—The thermostated forced-air bath is generally accepted as the best practical method of temperature regulation for most applications. Temperatures can be controlled by regulators or proportionally controlled heaters using a thermocouple or platinum-resistance thermometer as a sensing element. The advantage of a forced-air bath is the speed of temperature equilibration. Air bath ovens are readily adaptable to temperature programming and are capable of operating over a range of 35 to 450°C. This range can be extended down to −100°C by using cryogenic equipment.

6.2.2 *Other Devices*—Liquid baths, drying ovens, incubators, or vapor jacket enclosures are less stable, less convenient means of providing a source of heat to maintain or raise the temperature of a chromatographic column. These devices are not recommended for precision chromatographic applications.

6.3 *The Injection Port*—The purpose of the injection port is to introduce the sample into the gas chromatographic column by instantaneous volatilization following injection into the gas chromatographic system. Two sample inlet types are in common use in gas chromatography: the flash vaporization and the on-column injection inlets.

6.3.1 The temperature of the flash vaporization inlet should be above the boiling points of the sample components and is limited by the amount of septum bleed generated and the temperature stability of sample components. It should be set at that temperature above which no improvement in peak shape occurs but should be determined by the nature of the sample and the volume injected, not by the temperature of the column. If the inlet temperature is too low, broad peak with a slowly rising front edge will result from slow vaporization of the sample. If the temperature is set far above what is necessary to produce fast vaporization, thermal decomposition of the sample, decreased septum life, and ghost peaks due to septum bleed may be observed. Generally, a good guideline is to maintain the inlet temperature 25 to 30°C higher than the highest boiling point of any sample component.

6.3.2 A glass liner placed inside the injection port will eliminate sample contact with hot metal inner walls of the inlet, which can catalyze thermal decompositions. Any debris left in the liner, especially from biological samples, can be a source of excessive sample adsorption. If a liner is used, the debris can easily be removed by replacing the liner. Deactivation of the glass liner by treatment with dimethyldichlorosilane may be necessary for some compounds.

6.3.3 With on-column injection technique, the sample is deposited in the liquid state directly on the column packing. The sample must be small enough to preclude flooding of the column, with possible detrimental effects to peak shape and column life. Ideally, the on-column inlet is a part of the column, so its temperature may be controlled as the column temperature is controlled. In practice, because an on-column inlet usually has a somewhat higher thermal mass than an equivalent sector of the rest of the column, the inlet must be heated somewhat above the maximum analysis temperature of the column oven. The criteria of good peak shape and quantitation should be used to determine the maximum required temperature for the inlet. One should consider the temperature limit of the column packing when heating the injection inlet and detector. With some samples, a nonheated injection port is adequate, especially with temperature-programmed operation.

6.3.4 *Injection Port Septum:*

6.3.4.1 The septum is a disc, usually made of silicone rubber, which seals one end of the injection port. It is important to change the septum frequently after two to three dozen injections, or preferably at the end of the working day. The best technique is to change the septum when the column is relatively cool (below 50°C) to avoid contact of stationary phase in a hot column with air (danger of oxidation). After the septum is changed, return the inlet temperature to that which was originally set. The inlet temperature should be the optimum for the particular analysis, as well as within the recommended operating temperature of the septum. If the septum is punctured too many times, it will leak air into the gas chromatographic system, even though it is under pressure. At high temperatures, above 150 to 200°C, air (oxygen) in the carrier gas from a septum leak will degrade the stationary phase. An excessive septum leak will also produce a change in carrier gas flow rate (a change in retention time) and loss of sample (irreproducible peak heights) due to outflow from the leak. When installing the septum, do not overtighten the retaining nut. The septa will swell at high temperature and extrude out of the injection port. A snug fit at room temperature is sufficient. It is important for septum life to make sure the injection needle is sharp with no bent tip. Fine emery cloth, or a fine sharpening stone, can be used to sharpen the point.

6.3.4.2 Ghost peaks may be observed in temperature programmed runs due to septum bleed. Septum bleed is due to the thermal decomposition, 300°C or higher, of the septum that produces primarily lower molecular weight cyclic dimethylsiloxanes. It contributes to baseline response and is frequently observed as evenly spaced peaks in a temperature programmed run in which no sample has been injected. This situation can be demonstrated by the disappearance of ghost peaks after placing aluminum foil (precleaned with solvents such as methylene chloride or toluene) over the inner face of the septum or by turning off the injector temperature and making several blank runs. Septum bleed can be decreased by using either air- or water-cooled septum retaining nuts, by using a septum flush head, or by using special high-temperature septa which are available from a number of gas chromatographic supply houses.

6.4 *Detector Temperature Control*—The detector temperature should always be above that of the maximum operating analytical temperature, to prevent the possibility of condensation of sample components or stationary phase bleed in the detector and connecting line. Because there is usually some temperature gradient across a detector, the temperature should be set at 30 to 50°C above the maximum analysis temperature to ensure that the entire detector is hot enough to prevent condensation. Usually, it is neither necessary nor desirable to use an excessively high temperature since this can result in reduced sensitivity, increased noise level, frequent need to clean the detector, and thermal decomposition of sample or stationary phase.

6.5 *Measurement of Temperature*—The choice of sensing elements used to measure temperature depends on the desired accuracy (control about a set point) and precision of the measurements. Instrument read-outs should be verified periodically. Some common temperature measurement devices are as follows:

6.5.1 *Standardized Mercury Thermometer:*

Range, °C	Accuracy, °C
0 to 100	±0.02
100 to 200	±0.05
200 to 400	±0.50

6.5.2 *Calibrated Platinum Resistance Thermometer:*

Range, °C	Accuracy, °C
−140 to 500	±0.01

6.5.3 Thermocouple (iron – constantan, or other).

6.6 *Analytical Column:*

6.6.1 The analytical column is a length of tubing (glass, metal, or plastic) that is filled with a packing material. It is discussed thoroughly in Section 8.

6.6.1.1 *Column Characteristics*—Specified by method.

6.6.1.2 *Carrier Gas*—Specified by method.

6.6.1.3 *Sample Size*—Variable within limits.

6.6.1.4 *Flow Rates of Carrier Gas and Detector Gases*—Variable within limits.

6.6.1.5 *Column Temperature*—Usually specified by method, and

6.6.1.6 *Physical or Chemical Characteristic of Compound Analyzed*, or both.

6.6.2 *Detector Characteristics*—Desirable detector characteristics should include the following:

6.6.2.1 Good stability (low noise level, minimum response to changes in temperature and flow rate).

6.6.2.2 Ruggedness and simplicity.

6.6.2.3 Sensitivity to the components for which analysis is desired. Use either a selective detector for materials of interest or one with a similar response for all components.

6.6.2.4 Linearity of response versus sample concentration. Wide linear range.

6.6.2.5 Rapid response to changes in column effluent composition (small internal volume or flow-through design, or both).

6.6.2.6 Detectors, which are nondestructive and do not contribute to band broadening may be used in series with other detectors.

6.7 *Types of Detectors*—The detector is located at the outlet end of the chromatographic column and both senses and measures the amount of components that have emerged from the column. The optimum detector should have high sensitivity, low noise level, a wide linearity of response, a response to all compounds of interest, and yet be insensitive to changes in flow and temperature. Selective detectors are characterized as having selective, or greatly enhanced response to certain components. Linearity is decreased for all detectors by column bleed. As many as forty detection systems have been reported, yet only about a dozen are commonly used. Table 1 shows some of the more commonly used detectors. Of these, the thermal conductivity, the flame ionization, the electron capture, the nitrogen-phosphorus, and the flame photometric detectors are the most popular. Nondestructive detectors should be vented to a hood to remove any toxic effluents from the workplace. The effluent from destructive detectors may also be toxic. Details on detectors can be found in the applicable methods in Practices E 516, E 594, E 697, E 840, and E 1140.

6.8 *Programmed Temperature Operation*—The apparatus used in programmed temperature gas chromatography differs in some respects from that normally used for isothermal work. Basically, the column temperature is varied with time (program rate) to enhance speed of separations. The advantages of using programmed temperature operation include better resolution of lower boiling components because of lower starting temperature and greater sensitivity because of sharper peaks for the higher boiling components.

6.8.1 *Column Heater and Temperature Programmer*—It is of utmost importance that the column temperature program be reproducible, and that the difference between the set (desired) temperature and the true average column temperature be as small as possible. However, these requirements are difficult to achieve at high heating rates and with columns of large diameter. The mass of the column and its heater should be kept as small as possible. This will minimize thermal lag and will give proportionately small variations around the set temperature at any time. Proportional temperature controllers supply almost full power to the heater until the set point is very closely approached.

6.8.2 The recirculating air bath is the recommended method of heating in programmed temperature gas chromatography (PTGC). The obvious advantages are extremely rapid heating (and cooling after an analysis is completed) with very little temperature lag.

6.8.3 Liquid baths may be used for very low heating rates. They are commonly contained in taped Dewar flasks.

6.8.4 No matter what type of heating device is used, accurate control of the temperature program is necessary. This is usually accomplished by appropriate electronic systems that develop linear (or other) programming rates as desired.

6.8.5 Detectors for programmed temperature gas chromatography should be relatively insensitive to minor temperature and flow fluctuations and insensitive to stationary-phase bleed. These difficulties can be overcome by operating the detector at or near the upper temperature limit for the analysis and by using adequate flow controllers. If stationary-phase bleeding is excessive during PTGC runs, a second conditioning procedure (Section 9) might improve the situation. Alternately, a duplicate analysis column can be used on the reference side of the detector. By equalizing substrate bleed on both sides of the detector, the baseline drift can be substantially compensated. However, this technique does not improve column life and is detrimental to detector linearity. If at all possible, operate the column within its recommended temperature range.

6.8.6 When using the temperature programming technique, the resistance to carrier gas flow in the gas chromatographic column increases with increasing temperature. The flow controllers need a positive pressure of 10 psi to operate properly. By setting the second stage of the regulator to 40 to 60 psi, there will usually be sufficient excess pressure to maintain a constant gas flow through the column. Higher pressures might be required to maintain flow when using relatively long columns of 10 ft, or longer, or packings finer than 120 mesh.

7. Hazards

7.1 *Gas Handling Safety*—The safe handling of compressed gases and cryogenic liquids for use in chromatography is the responsibility of every laboratory. The Compressed Gas Association, a member group of specialty and bulk gas suppliers, publishes the following guidlines to assist the laboratory chemist to establish a safe work environment: CGA P-1, CGA G-5.4, CGA P-9, CGA V-7, CGA P-12, and HB-3.

TABLE 1 Applicability of Commonly Used Gas Chromatographic Detectors

Detector[A]	Applicability (Type of Compound)	Range of Minimal Detectable Amounts (grams)
Thermal Conductivity	All	10^{-6} to 10^{-7}
Flame Ionization	Organic	10^{-12}
Electron Capture	Halogenated and Oxygenated	10^{-12} to 10^{-15}
Flame Photometric	Sulfur, Phosphorus	10^{-11}
Alkali Flame	Nitrogen, Phosphorus	10^{-12} (even lower for phosphorus)

[A] Further information can be found in Practices E 516, E 594 and E 697.

8. Materials

8.1 *Stationary Phases*—The stationary phases (partitioning agents) that have been successfully used for specific separations are found most quickly by a literature search. Many phases are listed in ASTM publications AMD-25A and AMD-25A-51.[4] The most desirable stationary phases do not volatilize (bleed) significantly from the solid supports at temperatures required to elute the sample.

8.1.1 The polarity of the stationary phases is currently best characterized by McReynolds Constants.[5] The higher the McReynolds Constant, the more polar the phase. Rohrschneider constants can also be used to measure the polarity of stationary phases.[6]

8.1.2 The effects of using polar and nonpolar stationary phases are summarized as follows:

8.1.2.1 Nonpolar stationary phases separate compounds primarily by order of relative volatility or boiling point.

8.1.2.2 Polar stationary phases separate compounds by order of both relative volatility and relative polarity. With polar phases, nonpolar compounds will elute before polar compounds of the same boiling point.

8.1.2.3 Polarity alone is insufficient to describe the separation power of a column. One must consider the overall selectivity of a column towards a set of analytes. This selectivity is a summation of the effects of dispersive interactions, acid-base interactions and the dipole interactions offered by the various pendent groups in the stationary phase.

8.1.3 The stationary phases used in gas chromatographic columns have both minimum and maximum temperature limits. The chromatographer must be aware of the limits for the phase being used. Below the minimum temperature, the phase will behave as either a viscous liquid or a solid. Less efficient separation will be observed, and the chromatographic results will be exhibited as broader peaks in the gas chromatogram due to poor mass transfer of components in the stationary phase.

8.1.3.1 Above the maximum temperature limit, the phase will begin to bleed off the column at an accelerated rate, and the observed results will include a drifting baseline or excessive spiking on the baseline. Under these conditions, the liquid phase will decompose or volatilize, and thus be removed from the column. This situation will eventually lead to decreased retention times with broader peaks resulting in poorer resolution of very close peaks. Peak tailing will also be observed as the uncoated surface becomes exposed by removal of liquid phase, thus shortening column life. Bleeding also can expose bare support surface that can adsorb molecules being analyzed and reduce column efficiency. In extreme cases, phase bleeding will result in fouling the detector and connecting lines. The observed maximum temperature will depend upon many experimental variables, such as type of liquid phase column, conditioning, phase-loading level, column temperature, sensitivity setting of the detector, and purity of the carrier gas. In programmed temperature runs, the column can sometimes be operated for short periods about 25°C above maximum temperature. However, column bleed should be minimized for quantitative results since it decreases the linear range of all detectors.

8.2 *Active Solids:*

8.2.1 *Molecular Sieves*—The synthetic zeolite molecular sieve sorbents separate molecules by size and structural shape. Isomers with a more round shape, as branched versus straight chain molecules, diffuse in and out of the zeolite structure more easily than isomers with the long chain structures. Separations are affected by the differences in times required for molecules of different sizes to find their way into and out of the sieve-like structure of the adsorbent. Molecular sieves are most useful for separating H_2, O_2, N_2, CO, and CH_4. Carbon molecular sieves are also available, and can be used to separate O_2, N_2, CO, CO_2, H_2O, and C_1 to C_4 hydrocarbons.

8.2.2 *Porous Polymers:*

8.2.2.1 One type of porous polymer used in gas chromatography is available in the form of microporous cross-linked, polymer beads produced by copolymerizing styrene and divinylbenzene or more polar copolymers, or both. These materials are generally used as received without coating with any liquid phase. They provide symmetrical peaks for polar, hydrogen-bonding compounds such as water, alcohols, free acids, amines, ammonia, hydrogen sulfide, etc., and organic compounds up to molecular weights corresponding to about 170.

8.2.2.2 Another porous polymer is poly(2,6-diphenyl-*p*-phenylene oxide). This material is useful for the analysis of amines, alcohols, and hydrogen-bonding compounds. It is also used as an adsorbent for trapping trace organic compounds in water and air.

8.2.3 *Silica Gel, Alumina, and Carbon*—Among the active solid adsorbents are silica gel, alumina, and activated carbon. They are useful for low-boiling hydrocarbons.

8.2.4 Solid adsorbents modified by low concentrations of liquid phases may retain the advantageous properties of both. Some solid adsorbents can be modified by the addition of surface activating compounds such as wetting agents, silver nitrate, and the metal salts of fatty acids.

8.3 *Diatomaceous Earth Supports*—The most popular gas chromatographic supports are those prepared from diato-

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[4] *Gas Chromatographic Data Compilation*, ASTM, 1981.

[5] McReynolds, W. O., *Journal of Chromatography Science*, Vol 8, 1970, p. 685.

[6] Supina, W. R., and Rose, L. P., *Journal of Chromatography*, Vol 8, 1970, p. 214.

maceous earth, also called diatomaceous silica or kieselguhr. The two main types are white and pink in color. The white supports are recommended over the pink supports because of their more inert surface. The former are, however, very friable and must be handled very carefully when preparing packings and loading into gas chromatographic columns. Before using these supports, check the manufacturer's literature for comments on their use.

8.3.1 The white-colored supports are produced by calcination of diatomaceous earth with sodium carbonate as a flux. In this process, the diatomaceous earth fuses, due to formation of a sodium silicate glass. The product is white in color due to conversion of iron oxide into a colorless complex of sodium iron silicate. These white materials are used to prepare the more inert gas chromatographic supports. However, they are fragile and subject to abrasion from excessive handling in the course of sieving, packing, or shipping. Abrasion will produce finer particles, or fines, which will decrease column efficiency.

8.3.2 The pink-colored supports are prepared by crushing diatomaceous earth firebrick that has been calcined with a clay binder. The metal impurities remaining form complex oxides that contribute to the pink color of the support. These pink supports are denser than the white supports because of the greater destruction of the diatomite structure during calcination. They are harder and less friable than the white supports and are capable of holding larger amounts of liquid phase (up to 30 %) without becoming too sticky to flow freely. Their surface is generally more adsorptive than white supports. For this reason, they are not recommended for use in the gas chromatographic analysis of polar compounds. However, pink supports provide excellent efficiencies for the analysis of hydrocarbons and organic compounds of low polarity.

8.3.3 *Chemical Treatment of Diatomaceous Earth Supports*—Neither the pink nor the white materials give generally acceptable analysis of more polar compounds without further treatment. With these compounds, severe peak tailing is often observed, especially with the dense pink supports. This tailing is due to the presence of adsorptive and catalytic centers on all diatomaceous earth supports. The adsorptive sites are attributed to metal oxides (Fe, Al) and surface silanol groups, -SiOH, on the support surface. The latter are capable of forming hydrogen bonds with polar compounds.

8.3.3.1 Metal impurities are removed by washing with hydrochloric acid, which leaches out iron and aluminum and renders the surface both less adsorptive and less catalytically active. However, even with acid washing, the pink supports are still more adsorptive toward polar compounds than the white-type supports. Acid washing is sometimes followed by base washing, which seems to remove only minor amounts of metal impurities, but is a good pretreatment for supports that are to be used for the analysis of basic compounds.

8.3.3.2 Neither acid or base washing is effective in reducing peak tailing due to hydrogen bonding with the surface silanol groups, -SiOH. These groups are most effectively masked by treatment with dimethyldichlorosilane.

8.3.4 Acid-washed silanized grades of white diatomaceous earths are recommended as supports for nonpolar and medium polarity liquid phases. Because of the hydrophobic character of a silanized diatomaceous earth, even coating of the most polar liquid phases is difficult to achieve. Acid-washed, silanized grades of white diatomaceous earths are recommended as supports for the polar liquid phases, such as polyesters and silicones of high cyano-group content.

8.3.5 If the column is 6 ft (2 m), or less, use particle size of 100 to 120 mesh (125 to 149 μm) for highest efficiency under isothermal conditions. If the column is longer than 6 ft, use 80 to 100 mesh (149 to 177 μm) particles. If temperature programming is used, 80 to 100 mesh particles should be used to lessen resistance to carrier gas flow.

8.3.6 Further information concerning the liquid phase loading is given in 9.3.

8.4 *Halocarbon Supports*—The two types of halocarbon supports are those prepared from poly(tetrafluoroethylene) and poly(chlorotrifluoroethylene). These supports are relatively inert and are nonpolar. They eliminate peak tailing observed in the analysis of organic compounds capable of hydrogen bonding, such as water, alcohols, amines, etc. They are the preferred supports in the analysis of corrosive halogen compounds such as HF, BCl_3, UF_6, $COCl_2$, F_2, and HCl.

8.4.1 Poly(tetrafluoroethylene) supports require special handling procedures. When used as received, they are soft and tend to form gums upon handling. They can also build up a static charge and spray out of the column during the packing operation. These problems can be virtually eliminated by cooling the support to 0°C before coating with liquid phase and by avoiding the use of glass vessels. Rinsing poly(tetrafluoroethylene) with methanol and drying before use is another way to eliminate the static-charge problem.

8.4.2 Supports prepared from poly(chlorotrifluoroethylene) are structurally harder and are much easier to handle and to pack into a column.

8.5 *Tubing Materials*—Tubing materials should be chosen on the basis of the following criteria:

8.5.1 They should be nonreactive with the stationary phase, sample solvent, and carrier gas.

8.5.2 They should possess physical properties to withstand temperature and pressure of operating conditions, and

8.5.3 They can be shaped to fit in the column oven of the chromatograph.

8.5.4 Satisfactory materials include glass, nickel, stainless steel, and glass-lined stainless steel. Glass is the material of choice, unless conditions prohibit its use. Nickel tubing is more inert than stainless steel in most applications. Less frequently used column materials are poly(tetrafluoroethylene), aluminum, and copper.

8.6 *Carrier Gas*—The use of an impure carrier gas will produce problems in gas chromatography. Trace water and oxygen can cause decomposition of the liquid phase coated on the support. The common carrier gases, helium and nitrogen, should contain less than 5 ppm water and less than 1 to 2 ppm oxygen by volume. An oxygen adsorption trap can be used to remove trace oxygen, while trace amounts of water and hydrocarbons with molecular weights higher than methane, can be trapped on a molecular sieve trap. Place the molecular sieve drier nearest the gas supply. Calcium sulfate has been used in drying tubes, but cannot dry carrier gas to the same level as molecular sieve.

8.6.1 For some applications, hydrogen may be the preferred carrier gas. However, additional safety precautions are required due to hydrogen's explosive nature.

8.6.2 Air (oxygen) can leak into the gas chromatographic system through loose fittings or a septum, that has been punctured too many times, even though the carrier gas is under a pressure of 40 to 60 psi. Keep all fittings on the gas delivery lines tight, and check them at periodic intervals. Change the septum in the injection port frequently. Plastic tubing should never be used for carrier gas, hydrogen fuel (for FID), or make-up gas lines due to the possibility of oxygen or moisture diffusing through the tubing wall.

8.6.3 Each cylinder of carrier gas has its own impurity level. Occasional tanks contain large amounts of impurities which might overcome a low-capacity oxygen adsorption trap and destroy a gas chromatographic column at high temperature. A new tank or a fresh oxygen adsorption unit, or both will improve this situation.

8.6.4 Always change the tank when the pressure is less than 200 psi. As the total pressure in the cylinder decreases, there is an increase in the partial pressure of the water and other impurities adsorbed on the inner walls of the gas cylinder. As a result, the last amounts of gas delivered from the gas cylinder contain high levels of impurities.

8.6.5 *Carrier Gas for Instruments with Thermal Conductivity Detectors*—A major factor in sensitivity is the difference in thermal conductivity of the compound being analyzed and the thermal conductivity of the carrier gas. Helium (thermal conductivity = 33.60 cal/cm-s-°C) is usually the carrier gas of choice.

8.6.6 *Carrier Gas for Instruments with Flame Ionization Detectors*—The most commonly used carrier gases are nitrogen or helium. A maximum impurity level of 0.05 volume % does not generally interfere with most applications. Hydrogen is less commonly used in the US but is more popular in Europe because of availability and relatively low cost.

Note 2—If hydrogen is used, special precautions must be taken due to its explosive nature, to ensure that the system is free from leaks and that the effluent is properly vented.

8.6.7 *Carrier Gas for Instruments with Electron-Capture Detectors*—Users should follow the manufacturers' recommendations for the choice of carrier gas. Some common ones are nitrogen or 95 % argon/5 % methane. When using a tritium source in the detector, do not use hydrogen as the carrier gas. Hydrogen will replace tritium in the source.

9. Preparation of Packed Gas Chromatographic Columns

9.1 *Preparation of the Tubing Material:*

9.1.1 Glass columns should be cleaned and deactivated, first by rinsing with 30 mL acetone and then 30 mL toluene. Next, fill the column with 10 volume % solution of dimethyldichlorosilane in toluene. Allow the solution to stand in the column for 30 min. Finally, rinse the column with anhydrous toluene and then anhydrous methanol to cap unreacted DMDCS CL groups. Dry the column by passing a stream of dry nitrogen through it. Cap both ends of the column until such time that it can be packed.

9.1.2 Metal columns should be cleaned thoroughly before packing by rinsing with methanol, acetone, and chloroform. The column should be dried by passing nitrogen or dry air through it. Do not blow house air through the column since this compressed air usually contains an oil aerosol from the pump.

Note 3—Most chromatographic supply houses provide metal tubing that has been washed with solvents and is ready for use.

9.1.3 An alternative procedure is recommended for nickel tubing and can be used to clean stainless steel tubing. Rinse the nickel tubing with ethyl acetate, methanol, and distilled water. Then fill the tube with 20 volume % nitric acid and let it stand for 10 min. (**CAUTION:** Work in a hood and wear safety equipment when using nitric acid.) Next, rinse the tube with distilled water to neutrality and then rinse with methanol and acetone. Finally, dry the column by blowing nitrogen or helium through it.

9.1.4 The column length is generally 3 to 6 ft (1 to 2 m). Shorter columns can be used to decrease the time of analysis or to separate high boiling compounds. Longer columns are used to improve resolution, but have longer analysis times. (Columns longer than 20 ft (6.1 m) require excessive pressures to maintain the proper carrier gas flow.) A compromise is usually made between analysis time and resolution. As a general rule, an increase or decrease of column length by a factor of 3 to 4 is necessary to see a significant change in peak separation.

9.1.5 The diameter of the column can be ⅛ in. (3.2 mm) or ¼ in. (6.4 mm) outside diameter. The ⅛-in. column has less sample capacity, but greater efficiency, and is the most common type. Glass columns are generally 2 mm or 4 mm inside diameter. Some analysts have found that 3⁄16 in. (4.8 mm outside diameter) metal columns are the ideal combination between the capacity of ¼ in. (6.4 mm outside diameter) columns and the efficiency of ⅛ in. (3.2 mm) outside diameter columns.

9.2 *Choice of Diatomaceous Earth Support for Packed Columns*—See 8.3.

9.3 *Phase Loading on Diatomaceous Supports*—For preparative work and analysis of substances boiling below room temperature, use 15 wt % loadings for white-type supports and 30 wt % for pink-type supports. For general work, use loadings of the range of 3 to 15 wt %. For highest efficiency, shortest retention times, and the least amount of bleed during high-temperature operation, use 3 wt. % loadings. The lower phase loadings have lower sample capacity and elute components more rapidly and at lower temperatures. Always check the manufacturers' literature for suggested phase loadings for a particular support. For some applications (especially headspace analysis) loadings as low as 0.2 wt. % are used which result in very narrow peaks and short analysis times. High phase loadings tend to produce less reactive packings.

9.4 *Preparation of the Gas Chromatographic Packing*—The following procedures describe the coating of a solid support with stationary phase. The following four methods are commonly used to prepare gas chromatographic packings: (*a*) Filtration or Solution Coating Method, (*b*) Rotating Evaporator Method (*c*) Evaporative Method, and (*d*) Vacuum Evaporative Method. When preparing packings with loadings in the range of less than 5 wt %, the Filtration or Solution Coating Method is recommended. This method is preferred because it provides minimum handling of the friable white-type supports. For loadings of more than 5 wt. %, other methods can be used. The Rotating Evaporator

Method is recommended, but should only be used if a rotating evaporator is available, which turns very slowly at 20 to 30 rev per min.

NOTE 4—A 5 wt. % loading of stationary phase consists of 5 g stationary phase added to 95 g of support.

9.4.1 *Filtration or Solution Coating Method*—Prepare 100 mL of a solution of the desired phase in a vacuum filter flask. Use a suitable high boiling solvent (boiling point more than 60°C). The actual loading of the liquid phase on the support will depend upon both the viscosity of the phase solution and the density and mesh size of the support.

9.4.1.1 Add 20 g of support to the filter flask. Reduce the pressure in the filter flask for a few minutes with a water aspirator, then release the vacuum. Repeat this procedure for several cycles in order to remove air bubbles from the pores of the support particles. Be prepared to release the vacuum if the slurry foams excessively.

9.4.1.2 Allow the slurry to stand for several minutes. Pour the slurry into a coarse-frit sintered-glass filter funnel, and allow the solvent to drain freely until the support settles.

9.4.1.3 Apply vacuum cautiously and stop instantly when the solvent stops dripping. Dump the support into a flat borosilicate glass dish, and allow it to dry. Do not scrape the particles out of the funnel, since this might crush the particles. Do not resieve before use.

9.4.1.4 The actual phase loading will depend upon the viscosity of the phase solution and both the density and particle size of the support. For example, a 2 % solution of dimethyl silicone gum liquid phase will give a 3.8 wt % loading on white-type supports. A 10 wt % solution of a less viscous liquid phase will give a 5.5 wt % loading on white-type supports and 7.5 wt % on pink-type supports. Loadings obtained with other phases on other supports are best determined by experimentation.

9.4.1.5 The best way to determine the percent loading is to extract it from the support by extraction in a Soxhlet apparatus and determine the weight loss. Alternatively, measure the volume of solution recovered and calculate the volume of solution held up by the support. Calculate the approximate percent loading on the support by assuming that the concentration of the solution does not change.

9.4.2 *Evaporative Method:*

9.4.2.1 Weigh out the desired amounts of support and phase. Use a larger amount than that required to account for attrition, spills, etc. Dissolve the liquid phase in a chemically inert, low-boiling solvent contained in a filtration flask (see Table 2). (Most catalogs of gas chromatography equipment suppliers contain lists of suitable solvents.)

9.4.2.2 Gradually add the support to the solution with gentle swirling or agitation but with no mechanical stirring. (Suggested solvents are given in Table 2.) The amount of solution should be just enough to wet the solid support and form a slurry with little excess solvent.

9.4.2.3 Evacuate the flask briefly several times to remove air bubbles from the pores of the support. Be prepared to release the vacuum if the slurry foams excessively.

9.4.2.4 Transfer the slurry to a large flat borosilicate glass dish, and slowly evaporate the solvent in a hood with no further handling. The dish must be of a size that the packing is spread on the bottom in a thin layer, no more than about ¼-in. thick. A borosilicate glass baking dish makes a suitable container.

TABLE 2 Solvent for Liquid Phases

Liquid Phase Type^A	Solvent
Dimethyl Silicone	Toluene
Phenylmethyl Silicone	Ethyl Acetate
Cyanopropylphenyl Silicone	Methylene Chloride
Trifluoropropyl Silicone	Ethyl Acetate
Polyethylene Glycol	Methylene Chloride
Cyanopropyl Silicone	Methylene Chloride
Other Liquid Phases	Use solvent as recommended by supply house.

9.4.2.5 The critical stage occurs when excess solvent has evaporated, but the bed is still quite damp with a slight excess of solvent. Break up the damp bed by gently raking it with a spatula. As the solvent evaporates from the surface of a static bed of support, it leaves a higher concentration of phase at the bed surface. Therefore, the bed must be broken up frequently during the final stages of solvent evaporation to prevent formation of an unevenly coated support.

9.4.2.6 Continue to air-dry the material in the hood until the last traces of solvent are gone. Avoid excessive handling of the particles to prevent formation of fines due to abrasion, especially in the case of the white-type supports.

9.4.3 *Rotating Evaporator Method*—Prepare the slurry of support and phase as described in 9.4.2.1 to 9.4.2.3, except in an indented, round-bottom flask. Connect the flask to a rotating evaporator. Rotate the flask very slowly (less than 20 to 30 revolutions per minute) and evaporate the solvent under reduced pressure (water aspirator). Very slow rotation is necessary to prevent the particles from abrading against each other. Use of a heat lamp increases the evaporation rate. This method is not recommended for fluorocarbon supports.

9.4.4 *Vacuum Evaporative Method*—Prepare a slurry of support and phase in a filtration flask of sufficient capacity. (Suitable solvents are given in Table 2.) Attach the flask to a vacuum source (water aspirator) and apply vacuum briefly. (Be prepared to release the vacuum if the slurry foams excessively.) Repeat this procedure several times in order to remove the air bubbles from the pores in the support.

9.4.4.1 Apply the vacuum for a longer period, and swirl the contents of the flask occasionally until all the solvent is almost evaporated. This is the critical stage.

9.4.4.2 Now shake the contents of the flask by gently bumping the flask on a wood or plastic board. This will break up the bed of packing. Do not allow the solvent to evaporate from the surface of the support bed. Otherwise, the solvent will evaporate and leave a higher concentration of phase at the bed surface.

9.4.4.3 Continue to apply vacuum until the packing is a freely flowing powder, then transfer it to a tray for air-drying in a hood.

9.4.5 *Fluidized Drying Technique*—This technique has been used to produce efficient, uniformly coated packings. During the drying stages of methods 9.4.1 to 9.4.4, when the packing has reached the consistency of a wet sand, add it to a fluidizer. Then dry the packing by passing a flow of inert, warmed gas (nitrogen or helium) through the bed of packing.

9.5 *Packing the Gas Chromatographic Column*—The purpose in packing a gas chromatographic column is to fill the column with packing as completely as possible, leaving no empty spaces. Two variations are noted in 9.5.3 and 9.5.4 (a pressure-fill procedure and a vacuum fill procedure).

9.5.1 It is preferable to coil the column before packing to prevent crushing of the support particles. Metal columns can be coiled after loading to meet equipment requirements. Bends in the packed region must never be made with radii less than those specified in 9.5.2, to avoid crushing the packing in the column.

9.5.2 Right-angle bends are often necessary to make connections to injection and detection systems, and must be made before packing the column since some tubing deformation will occur, which will crush some of the solid support. Bends for such purposes should be within 4 in. (10 cm) of the column ends. For coiled columns, minimum diameter mandrels should be as follows: for 1/8 in. (3.2 mm) OD column use a 1 1/2-in. (38-mm) mandrel; for 1/4 in. (6.4 mm) OD column use a 2-in. (51-mm) mandrel. These configurations do not preclude the use of U- or W-shaped columns. If a U- or W-shaped column is to be used, the minimum 180° bend diameter must be at least that given for the above mandrel sizes.

9.5.3 *Pressure Fill Procedure*—To each end of the column to be filled, fit a nut, a back ferrule, and a suitable front ferrule. Place a small plug of silanized glass wool into the detector end of the column, and cap the column by screwing in a metal cap with a 1/16-in. vent hole drilled into it. When analyzing trace acidic compounds, as organic acids and phenols, adsorption can be decreased by using phosphoric acid-treated glass wool to plug the column ends. Wear safety glasses when pressure-packing columns.

9.5.3.1 Attach the end of the empty column to an apparatus similar to that shown in Fig. 2A. Add to the reservoir sufficient packing material to fill the column, plus about 30 %. Attach the upper end of the reservoir to a nitrogen supply line controlled to provide approximately 40 psi. Check that all connections are tightened, place a safety shield in front of the setup, and apply 40 psi to the system.

9.5.3.2 As the stationary phase starts to fill the column, gently tap the column with a wood rod (handle of spatula or screwdriver) or an electrical vibrator set at a very low vibration level. Continue tapping until the packing shows no voids and the level of packing in the reservoir remains constant.

9.5.3.3 Shut off the nitrogen supply and wait for the pressure to dissipate. Disconnect the column from the reservoir. Do not disconnect the column while it is under pressure. Have a clean beaker available to collect excess packing material that will fall from the opened reservoir. Tap out about 1/8 in. (3 mm) of column packing, and replace it with a silanized glass wool plug. Affix a metal column tag engraved with a description of the stationary phase, loading, support, and the assigned column number.

9.5.4 *Vacuum-Fill Procedure:*

9.5.4.1 Clamp the column so that the detector and injector ends point upward. Plug the detector end of the column with a 1/4-in. plug of silanized glass wool. Use phosphoric-acid treated glass wool when analyzing for trace organic acids and phenols.

9.5.4.2 Attach a small funnel to the injection port end of the column. Attach the detector end of the column to a vacuum source, either a vacuum pump (preferably) or a water aspirator. (If a water aspirator is used, a 500-mL filter flask, or the device shown in Fig. 2B, should be placed in the line between the pump and the column.) Do not turn on the vacuum yet.

9.5.4.3 Add 1 to 2 mL of packing to the funnel, and tap the column gently to settle the packing. A pencil or a wooden spatula handle can be used. Alternatively, the column can be stroked with a plastic saw. The use of an electric vibrator is not recommended. Excessive vibration will cause the particles to abrade against each other, producing fines and newly fractured surfaces that are not coated with stationary phase.

9.5.4.4 Turn on the vacuum source. Continue to add the packing in small increments with tapping until the column is full. Finally, apply pressure to the head of the column to pack it a little tighter. However, take care to make sure the pressure is equalized slowly, because packing will be blown out of the column if the pressure is released too suddenly.

9.5.4.5 Next, tap out enough packing to create a 1/8 in. (3 mm) void space at the injector port end of the column. Plug this end with a silanized glass wool plug. Do not pack the plug too tightly. This will either impede the carrier gas flow or crush the packing particles.

9.5.4.6 Higher efficiencies are always observed if the column is packed for on-column injection. In this technique, the column is packed so that there is space at the injection port end of the column, which is then placed inside the injection port. This void space should be of such a length that the injection needle just reaches, or slightly penetrates, only the glass wool plug, not the packing, when the column is installed. Thus the sample is injected almost directly onto the column.

9.6 *Conditioning of Packed GC Columns:*

9.6.1 The purpose of the conditioning process is to remove extraneous material (solvent and adsorbed material) from the column before analytical usage. Since the column is heated, the liquid phase also redistributes itself over the support surface to provide a more even coating.

9.6.2 Install the column into the gas chromatograph at the injection side only. Do not connect the column to the detector during the conditioning stage. Any column bleed might foul the detector and the connection lines between the column and detector. Turn on the normal analytical carrier gas flow and flush air out of the column at ambient temperature for 30 min.

9.6.3 Heat the column at a rate of 2°C/min to the conditioning temperature. The latter temperature should be at least 25°C higher than the analytical temperature but 25°C lower than the maximum operating temperature recommended for the liquid phase. Maintain this temperature overnight with carrier gas flow.

9.6.4 The next day cool the column and connect it to the detector. Detectors operated in very sensitive modes, particularly the electron capture detector, might require two or more days of conditioning at the higher temperatures before a satisfactory baseline is obtained. (Other sources for baseline drift and noise are impurities in the carrier gas, a dirty detector, air leaks in the gas-line fittings, insufficient carrier gas pressure, a much-punctured septum, chemical decomposition of the phase (due to presence of traces of acid or base on the support, in the phase, or on the inner column walls, and incorrect fuel gas ratios to the flame ionization detector.)

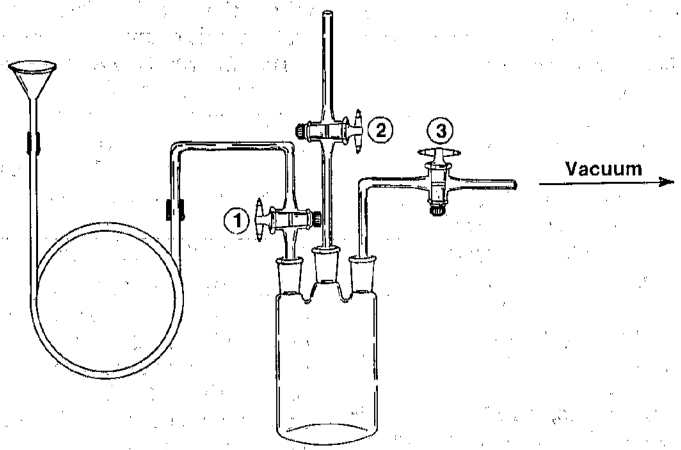

FIG. 2A Vacuum Fill Apparatus

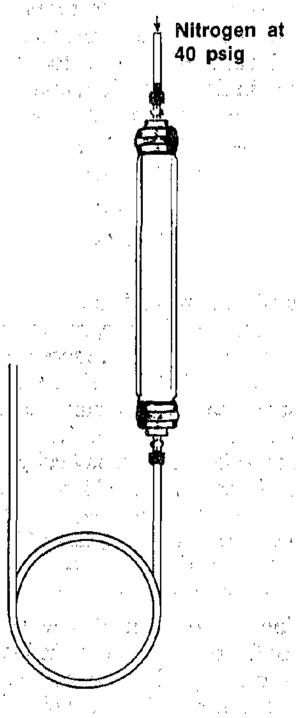

FIG. 2B Pressure Fill Apparatus

9.6.5 There is a special "no-flow" conditioning procedure which can be used with certain silicone phases, as methyl and methylphenyl silicones with or without low vinyl content. It has been reported to improve analysis for drug compounds. The procedure starts by conditioning the column for ½ h as described in 9.6.2. Turn off the carrier gas flow, cap the free end of the column with a metal cap, and heat at 310°C for 1.5 h with no carrier gas flow. Cool the column to 100°C. Uncap the oven, turn on the carrier gas flow, and continue the regular conditioning procedures listed in paragraph 8.6.3.

NOTE 5—This no flow conditioning procedure may damage or destroy non-silicone containing stationary phase or silicone containing functional groups other than phenyl or methyl.

9.6.6 Many of the liquid phases are commercial-grade material, and conditioning might require several days before the noise level is low enough to provide usable baselines at high sensitivity. The use of gas chromatographic-grade phases is recommended since they have been carefully purified and long periods of conditioning are usually not necessary.

10. Sample Injection Procedures

10.1 *Injection Technique*—Useful analyses are obtained only by injecting representative samples into the gas chromatograph. Since chromatographic samples are small, the best practices and procedures must be followed.

10.1.1 If elution is to be otherwise, the sample injection must be almost instantaneous in order to introduce the sample as a plug. Avoid unnecessary sample dilution and inadvertent trapping.

10.2 *Sample Size*—Sample sizes used for analysis with 2- to 5-mm ID packed columns are in the range of 0.1 to 10 µL multi-component liquids. Gas samples usually range from 10 µL to 2 mL.

10.2.1 There is frequently a reduction in retention times with sample size when the column is overloaded. Therefore, when identifying by comparison with a known sample, the same amount of each component must be used in an amount that does not overload the column. The sample size that overloads the column is that size which decreases the efficiency (Section 11) by 10 % compared to a smaller sample size. Sample overload is sometimes shown by peaks with sloping fronts and backs that are almost perpendicular to the baseline. (Another reason for this peak shape is insufficient vaporization of the sample. Either the injection port or the column temperatures, or both, are too low.)

10.3 *Sample Injection Devices*—Samples may be introduced by syringes, automatic sample injectors using syringes, or sample valves. (There are also devices that introduce capsules containing sample into the injection port.) For rigorous quantitative work, any sample introduction device should be flushed and filled at least three times with the sample immediately before use.

10.3.1 The most common method for liquids is the use of a syringe injection technique through a self-sealing septum. In the usual 10-µL syringe, there is approximately 0.8 µL of dead volume in the syringe needle. This volume is additional to the volume in the syringe barrel and can be measured by withdrawing the entire sample volume into the syringe barrel.

10.3.1.1 First, pump the sample back and forth vigorously in the syringe to wet the plunger and to remove air bubbles. Then withdraw the sample back into the syringe so that the entire volume can be read on the volume marks of the syringe barrel. When preparing the syringe for injection, never leave the sample solution in the needle. This technique will minimize sample boiling out of the needle when it is inserted into the hot injection port.

10.3.1.2 In an alternative procedure, called the solvent-flush technique, load the syringe in the following order: solvent, air, sample solution, and air, with only air remaining inside the needle. When the sample is injected, the solvent is the last to leave the syringe, and it rinses out sample residue in the needle.

10.3.1.3 Wipe the syringe needle off before injection. Insert the needle carefully through the GC septum, inject the sample at once, and withdraw the needle in one continuous motion.

10.3.1.4 Often the tip of the needle becomes bent, forming a fish hook that will tear the septum on subsequent injections. This can be detected by brushing the end of the needle gently over the end of a finger. A few strokes on a sharpening stone will remove the fish hook.

10.3.1.5 Syringes should be cleaned with a solvent that will remove all traces of contamination. Consult the manufacturer's literature for further information. Many chromatographic supply vendors sell suitable cleaning solutions and kits. Liquid sample valves, in both automated and manual versions, are also available.

10.3.1.6 Gas samples are most conveniently injected using gas-tight syringes. These devices are quite satisfactory for survey work because the sample size can easily be verified. However, the syringe needle is more likely to clog with pieces of septum material when gas samples are injected than when liquids are injected. If no chromatographic peaks are observed, test the syringe by injecting air into a liquid. If no bubbles are seen, unclog the needle with a wire or by filling the syringe without the needle and forcing solvent through the needle. However, repeatable results in gas analysis will only be obtained using gas sample valves that have a fixed sample loop. The latter valves can easily be automated.

10.3.1.7 A sealed, friable, or puncturable ampule containing a weighed amount of sample may be placed in the injection chamber. The ampule is physically broken to release the sample, which is then swept into the gas chromatographic column by the carrier gas.

TABLE 3 n-Alkanes Used for Column Evaluation at 200°C

1 m 10 % Dimethyl silicone C_{18}, C_{20}, C_{22}
3 m 10 % Dimethyl silicone C_{14}, C_{16}, C_{18}
1 m 10 % Phenylmethyl silicone C_{20}, C_{22}, C_{24}
3 m 10 % Phenylmethyl silicone C_{16}, C_{18}, C_{20}
1 m 10 % Cyanopropyl silicone C_{24}, C_{26}, C_{28}
3 m 10 % Cyanopropyl silicone C_{20}, C_{22}, C_{24}
1 m 10 % Polyethylene glycol C_{24}, C_{26}, C_{28}
3 m 10 % Polyethylene glycol C_{18}, C_{20}, C_{22}

10.4 *Sample Container*—Care must be taken in the design and construction of sample containers so that none of the components of interest are in any way changed or removed from the sample by reaction, diffusion, or adsorption. Stopcock grease and material desorbed from plastic cap liners are frequent sample contaminants.

11. Evaluation of Column Performance

11.1 Make a test mixture which contains a normal aliphatic hydrocarbon and the compound being analyzed, or one similar in structure. The aliphatic hydrocarbon peak should appear in the chromatogram near the second component in the test mixture. (Suggested aliphatic hydrocarbon mixtures are shown in Table 3.) The peaks of both components should be about the same size. The analytical conditions should be chosen so that both chromatographic peaks will appear about 4 to 6 times the retention time (distance) of the solvent peak. (If a selective detector is being used, choose a compound that will give a response to that detector.)

11.2 The different methods of determining column efficiency are shown in Equations 1, 2, and 3. Fig. 3 shows the measurements made on a chromatographic peak which are used to determine column efficiency.

$$N = 16 (t_R/w_b)^2 \tag{1}$$

$$N = 5.54 \; (t_R/w_h)2 \tag{2}$$

$$H = L/N \tag{3}$$

where:

N = number of theoretical plates,

t_R = retention time, or distance, measured in mm,

w_b = width of the peak at base, measured in mm, (Determined by extrapolating, as shown in Figs. 3a and 3b.)

w_h = width of the peak at one-half the peak height, h, all measured in mm, (See Note 6.)

H = height equivalent to a theoretical plate, HETP, and

L = length of the column in millimetres, or in centimetres.

NOTE 6—The peak width may be measured to the nearest 0.1 mm by a magnifying loupe fitted with a scale graduated in 0.1 mm increments. The peak to be so measured should be at least 10 mm wide; this is arranged by choosing an adequately large chart speed.

11.3 Equation 1 is often used. However, it involves an extrapolation of the baseline, shown in Fig. 3, which can be in error. The use of Eq 2 is preferred because the term, w_h can be determined directly from the chromatogram without extrapolation. However, the width of the recorder pen line can be variable which can lead to difficulties in determining the true value of the peak width. Use a pen that writes with a sharp, thin line. The peak width should be measured from the leading edge of one line to the leading edge of the other line, as shown in Fig. 3a. For accuracy, determine the peak width twice (see Fig. 3a), and use the average value to calculate "*N*". To further minimize errors in determining the

TABLE 4 General Optimum Values of Carrier Gas Flow Rates

Column Dimensions	General Optimum Carrier Gas Flow	
	Helium	Nitrogen
⅛ in. OD or 2 mm ID	30 mL/min	20 mL/min
¼ in. OD or 4 mm ID	60 mL/min	40 mL/min

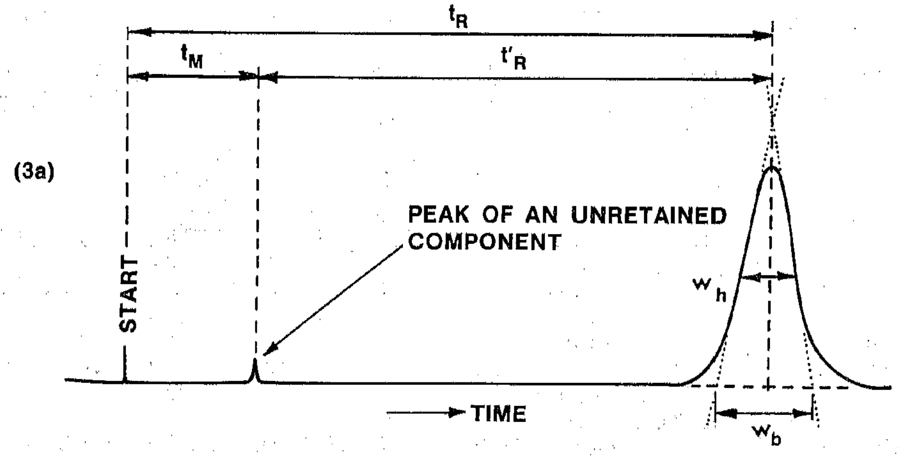

FIG. 3a Use of the Chromatogram to Calculate Column Efficiency

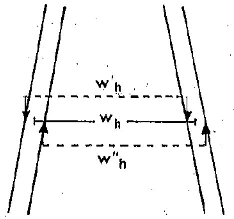

FIG. 3b Shows the Procedure to Measure the Peak Width, at w_h, to Account for the Thickness of the Pen. Use the Average of the Two Determinations, w'_h and w''_h, shown here

peak width, use a recorder chart speed that will give a minimum peak width of 4 to 5 cm. The peak height should be about 40 to 80% full-scale chart deflection. This can be achieved by either changing the detector sensitivity, or changing the sample size. However, be sure that the sample size does not exceed the capacity of the column.

11.4 The optimum efficiency occurs at the optimum carrier gas flow which can be determined by plotting the efficiency of the hydrocarbon peak versus carrier gas flow. General optimum values of the carrier gas flow rates are shown in Table 4, but should be determined for the particular column.

11.5 Calculate the efficiency of the second component. The results should be about the same for both components. If the efficiency of the second component is about 25 % lower than that of the hydrocarbon, the column might not be the best choice for analyzing the compound(s) of interest. A strong indication of nonsuitability is shown by comparatively greater tailing of the nonhydrocarbon component. Tailing can indicate strong interaction with the column packing (phase or support, or both), column tubing, or adsorption in the injection port or in lines leading from the column to the detector.

11.6 Typical efficiencies are shown in Table 5. Not all gas chromatographic packings are capable of excellent efficiencies. For example, porous polymers, Teflon supports, and some liquid phases, such as trifluoropropyl silicone can give efficiencies less than 500 plates per foot.

TABLE 5 Typical Efficiencies and Ratings of Gas Chromatographic Columns

Plates/ft	Plates/metre	HETP	Rating
500	1640	0.6 mm	Excellent
400–500	1300–1640	0.60–0.76 mm	Good
300–400	980–1300	0.76–1.0 mm	Fair
300	980	1.0	Poor

12. Use of the Gas Chromatographic Packed Column

12.1 Certain precautions and preventive maintenance are necessary to obtain the best column performance. Some of these points have been made before and will be referred to in this section. Further precautions will also be discussed.

12.2 *Carrier Gas Purity*—Trace, or adventitious, oxygen and water in the carrier gas can produce degradation of liquid phases on the support. Purification of the carrier gas and necessary precautions are discussed in 8.6.

12.3 *The Injection Port*—The injection port is often a source of trouble. The temperature can be either too low or too high. See 6.3. Another problem is a leaking septum. See 6.3.4.

12.4 *Column Care:*

12.4.1 Never heat a gas chromatographic column with air in it. When any column is first placed in a gas chromatograph, flush any air (oxygen) out of it by flushing with carrier gas at normal flow rates for 15 to 30 min. at ambient temperatures. Then heat the column to the desired operating temperature. Always cool the column to room temperature before removing it from the gas chromatograph. If the column is to be used again, cap the ends with metal caps to prevent diffusion of air (oxygen) into the column during storage. Contact of the stationary phase with oxygen when hot or for prolonged periods of time at room temperature cause degradation of the stationary phase. This is particularly the case with polyglycols and polyester type phases and to a lesser extent cyanosilicones. Other phases are affected to a varying degree.

12.4.2 After long periods of use, column performance may degrade as shown by peak broadening, tailing, or gradual merging of adjacent peaks. Often the problem lies in the front end of the column. The injection port temperature might have been too high and destroyed the initial section of the liquid phase on the column packing. Residues or decomposition products might have built up on the glass wool plug. These problems can be remedied by repacking the first few inches of a glass column, or cutting off the first few inches of a metal column. Use fresh silanized glass wool to close the end of the column in both cases.

13. Methods of Qualitative Analysis

13.1 Identification of compounds by gas chromatography alone cannot be absolute, and the results must be considered with care. Elution of a compound is dependent upon carrier gas flow rate, column temperature, support size, amount and type of liquid phase, column dimensions, instrument dead volume, and column pressure drop. These parameters must be stable to obtain reproducible results. The recommended format for a gas chromatographic method is given in Section 15.

13.2 Tentative identification of a compound can be made by comparing its adjusted retention time against those of known standards using exactly the same chromatographic parameters.

13.2.1 The retention time is the time interval measured from the point of injection to maximum peak height of the sample. Adjusted retention time, t'_R, is derived by subtracting the time required for an unabsorbed gas, like air, or methane, t_M, to traverse the column (also called the gas holdup time) from the retention time, t_R.

NOTE 7—On some solid adsorbent columns, such as molecular sieves, there is no nonadsorbed component.

$$t'_R = t_R - t_M \tag{4}$$

13.2.2 Retention times are affected by all chromatographic parameters. As a result, direct comparison of retention times of the same components on different instruments or between laboratories should be done with caution. Use of relative retention time is an easy practical technique for providing elution data. The retention of a component is expressed relative to the retention of a known reference standard. The reference standard should possess structural or chemical similarity to the compounds being analyzed.

13.3 The retention of a given weight of compound is usually independent of its concentration if the compound does not overload the column producing skewed peaks. The retention of the compound is also independent of other substances present if there is no appreciable overlap with another compound. Substances that exhibit positive, or Langmuir-type, skewing (tailing) during elution will produce a decrease in retention as the concentration increases; while negative, or anti-Langmuir-type fronting will produce an increase in retention time with increased concentration.

13.3.1 The logarithm of the retention time of members of a homologous series run isothermally is usually a linear function of the number of carbon atoms of a molecule. Using this characteristic, two or three reference compounds can provide sufficient information to prepare a plot of the logarithm of the retention time versus carbon number, and they can identify other members of the series. Retention time, adjusted or not, is of little value in comparing the results from various instruments. The use of Kovats retention index, based on the relative retention of a compound to the retention of normal paraffins, provides a more reliable means of comparing the results obtained from different instruments (see Practice E 355).

13.4 Absolute compound identification or characterization, must be made with ancillary techniques such as mass or infrared spectrometry, nuclear magnetic resonance, chemical analysis of the effluent, or spot tests for functional groups.

13.4.1 The samples for the analyses in 13.1 through 13.4 may be obtained by trapping components as they emerge from the chromatograph. A trap, glass capillary, or U-tube, is cooled with ice or dry ice, and placed in the effluent stream of the column. Several collections may be required to obtain a sufficiently large sample.

13.4.2 The collection of effluent is easiest with nondestructive detectors, see 6.7. In the case of destructive detectors, a split is made for the collection just before the detector.

13.4.3 Apparatus is also available so that the effluent from the gas chromatographic column can be analyzed directly by mass spectrometers or infrared spectrophotometers.

14. Methods of Quantitative Analysis

14.1 Gas chromatography can be used to determine quantitatively the composition of complex samples. There are several factors that must be considered before the sample is analyzed. The recommended format for gas chromatographic methods is given in Section 15.

14.1.1 *The Chemistry of the Sample*—The chemistry of the sample, if known, allows a chromatographer to select more accurately a column compatible with the sample and to anticipate potential interferences from reaction byproducts.

14.1.2 *The Choice of a Detector*—A detector must be chosen with the needed selectivity and sensitivity. If components will be analyzed at low levels, an electrolytic conductivity electron capture, nitrogen phosphorus, microcoulometric, ionization, or flame photometric detector should be selected. The detector may be limited to these lower concentrations and not applicable to high concentrations.

14.1.3 *Initial Separation of Components*—Next, a column must be chosen that will resolve the components of interest in the sample within a reasonable amount of time. First, a rough separation should be achieved with known standards. Next, actual samples should be analyzed to determine if there are any interferences. A second column, or an ancillary technique (GC/mass spectrometry, GC/infrared spectrometry, etc., should be used to verify that additional components are not eluting with the component of interest. Each new sample adds the possibility of an interference eluting with the component of interest; therefore this should be checked often. If an interference is detected, the chromatographer must change the method to remove it. The several options for doing this are as follows:

14.1.3.1 Select a column stationary phase with a greater selectivity for either the interference or the component of interest.

14.1.3.2 Choose a different type of detector that would detect the component of interest but not the interference. Examples would be water not being detected by a flame-ionization detector, or hydrocarbons not detected by an electrolytic conductivity or electron capture detector.

14.1.3.3 Consider other types of chromatographic separation such as capillary gas chromatography for more efficient separation of volatile compounds, liquid chromatography for separation of non-volatile compounds, or another appropriate separation technique.

14.1.4 *Detector Sensitivity and Linearity*—Once the chromatographic separation has been optimized, the detector can be optimized and calibrated. Gas flows should be adjusted to the optimum levels to get peak sensitivity at the concentration range of the components of interest. The detector must also be clean and leak-tight. (See the manufacturer's manual for suggested procedures.)

14.1.4.1 The linearity of the detector over the desired concentration range of the component(s) of interest is determined using prepared standards. This step will determine what the response is to increasing amounts of component. The peak area or height should be plotted versus the concentration for about five concentrations near the expected sample concentration. There should be a linear correlation. Nonlinearity may be caused by reactivity, adsorptivity, thermal sensitivity, or excessive column bleed. If the latter is the cause, change to a more thermally stable column or one of different polarity. Column reactivity can be characterized by skewed, misshaped peaks. This can be corrected by installing a fresh column of the same type that does not have reactive sites. Test mixtures can be used to demonstrate nonreactivity. Other sources of adsorptivity or reactivity with the sample are the injection port, connecting lines to the detector, or glass wool. Each of these sources can be detected by carefully troubleshooting the system.

14.1.4.2 The detector performance should be checked periodically throughout the analysis. This can be done by injecting one of the linearity standards and comparing it to the linearity plot.

14.1.5 *Peak Area or Height Measurement*—Many types of peak area and height measurement techniques exist. The oldest methods for calculating the peak area are manual measurement with a ruler of the peak area using one of the following equations:

$$\text{peak area} = w_h \times h \tag{5}$$

where:

w_h = peak width at half height, and

h = peak height

or

$$\text{peak area} = \tfrac{1}{2}\, w_b \times h \tag{6}$$

where:

w_b = peak width at the base of the peak, and

h = peak height.

Another precise measurement defines the peak area as retention distance (in millimetres) times the peak height (also in millimetres). For peak height, this distance is simply measured from the baseline to the apex of the peak. However, these techniques now, for the most part, have been replaced by electronic integration, which is much faster. The proper use of these devices is crucial for accurate quantitative analysis. The instruction manual for the particular integrator should be studied and understood thoroughly before attempting to use electronic integration for peak area or peak height measurement.

14.1.6 *Data Handling:*

14.1.6.1 All manufacturers supply an integral electrometer to allow the small electrical current changes to be coupled to recorders/integrators/computers. The preferred system will incorporate one of the newer integrators or computers that converts an electrical signal into clearly defined peak area counts in units such as microvolt-seconds. These data can then be readily used to calculate the linear range.

14.1.6.2 Another method uses peak height measurements. This method yields data that are very dependent on column performance and, therefore, not recommended.

14.1.6.3 Regardless of which method is used to calculate linear range, peak height is the only acceptable method for determining minimum detectability.

14.1.7 *Calibration*—It is essential to calibrate the measuring system to ensure that the nominal specifications are acceptable and particularly to verify the range over which the output of the device, whether peak area or peak height, is linear with respect to input signal. Failure to perform this calibration may introduce substantial errors into the results. Methods for calibration will vary for different manufacturers' devices but may include accurate constant voltage supplies or pulse generating equipment. The instruction manual should be studied and thoroughly understood before attempting to use electronic integration for peak area or peak height measurements.

14.2 *Types of Calculations:*

14.2.1 Each method of quantitative analysis has advantages and disadvantages. The four methods of quantitative analysis are as follows:

14.2.1.1 Internal standardization,

14.2.1.2 External standardization,

14.2.1.3 Normalization, and

14.2.1.4 Corrected area.

14.2.2 *Internal Standardization*—In this technique, a pure component (the internal standard) is added to a sample in a known amount. The peak area, or height, of all components of interest is compared to the peak area, or height, of the internal standard. These comparisons are referred to as response factors:

$$R_F = A_C/A_{IS} \times W_{IS}/W_C \tag{7}$$

where:

R_F = response factor,

A_C = peak area of component,

A_{IS} = peak area of internal standard,

W_{IS} = mass of internal standard, and

W_C = mass of component.

The amount of the component can be calculated from the weights of the sample and internal standard, the response factor, and the peak areas (or heights) as follows:

$$\%\ \text{Conc}_C = W_{IS}/W_S \times A_C/A_{IS} \times 1/R_F \times 100\ \% \tag{8}$$

where:

Conc_C = concentration of component in sample,

W_{IS} = mass internal standard,

W_S = mass sample,

A_C = peak area of component,

A_{IS} = peak area of internal standard, and

R_F = response factor.

This technique provides a correction for the relatively high variability of syringe injection and, therefore, yields a more precise method of analysis. Neither the quantity of solution injected, nor change in detector response, will alter the area ratio of the analyte and the internal standard. To achieve optimum performance, the internal standard must meet the following criteria.

14.2.2.1 The internal standard must elute in an area of the chromatogram that is free of sample components, or possible sample components.

14.2.2.2 The internal standard must not react with the sample or any of its components.

14.2.2.3 The internal standard and the sample must be homogeneous. A cosolvent may be used to produce a homogeneous mixture.

14.2.2.4 The internal standard must be easily and accurately added.

14.2.2.5 The internal standard must be pure.

14.2.2.6 The internal standard should elute near the component of interest.

14.2.2.7 The concentration of the internal standard, relative to that of the analyte, should be such that these two peaks are within 50 to 100 % of full scale deflection with the same electronic attenuation and sensitivity setting in order to allow manual measurements and calculations of parameters, if desired.

14.2.2.8 The most common use for the internal standard technique in chromatography is to correct for quantitative variations in the injection, particularly when using syringes. For this purpose, the internal standard need not be chemically related to the analyte, but must possess the criteria cited above and may be added in the final solution.

14.2.2.9 In certain applications, an internal standard with functional groups similar to the analyte may be desirable. For instance, those with a labile proton can be expected to exhibit similar adsorption isotherm behavior and to undergo similar physico-chemical transformations during such processes, as extraction from a complex matrix or derivatization, or both. Likewise, similar electronegative functional groups are likely to behave similarly towards an electron capture detector.

14.2.3 *External Standardization:*

14.2.3.1 This method compares peak areas or heights of components in a sample chromatogram to those in a standard solution injected separately. It is critical that accurate amounts of sample and standard be injected for the method to be valid. Generally, the solvent flush injection technique (see 10.3.1.2) or a sample valve of fixed volume is preferred.

14.2.3.2 The advantages of this method are as follows:

(*a*) Nondetected components do not bias the results.

(*b*) It can be used where several known components must be determined in a very complex sample.

(*c*) It can quantitate relatively reactive components.

(*d*) A single sample can be analyzed where maximum accuracy is not required.

(*e*) Nonlinearity has a minimal effect if the external standard is near the concentration of the sample.

14.2.3.3 The critical part of this method is the injection. The volume of sample in the injection syringe and standard must be accurately measured, allowing no bubbles in the slug of sample or standard solution. If the sample and standard have different densities, a correction must be made. Densities are easily determined by filling a 50-µL syringe to about 30 µL, wiping the needle, weighing it, expelling the sample, wiping the needle again, and reweighing it.

14.2.3.4 The peak areas or heights of the component in the sample and the standard compound are measured and the concentration calculated as follows:

$$\%\ \mathrm{Conc}_C = A_C/A_{ES} \times W_{ES}/W_S \times \%\ \mathrm{Conc}_{ES} \quad (9)$$

where:

Conc_C = concentration of component,
A_C = peak area of component in sample,
A_{ES} = peak area of external standard,
W_{ES} = mass of external standard injected,
W_S = mass of sample injected, and
Conc_{ES} = concentration of external standard in solution.

14.2.4 *Normalization*—This calculation assumes that every component elutes and that each has similar response factors. It is a fast procedure that requires no weighing. The sample is injected, and the peak areas or heights of all components are measured. The concentration of the component of interest is calculated as follows:

$$\%\ \mathrm{Conc}_C = A_C/A_{\mathrm{ALL}} \times 100 \quad (10)$$

where:

Conc_C = concentration of component of interest,
A_C = area of component, and
A_{ALL} = sum of areas of all components.

Severe errors result if the components have different response factors or do not all elute.

14.2.5 *Corrected Area*—This method corrects for differences in response but still assumes that all components elute and are observed by the detector. Response factors are used to correct for response differences as follows:

$$\%\ \mathrm{Conc}_C = A_C/(A \times R_F)_{\mathrm{ALL}} \times R_{F_C} \times 100\ \% \quad (11)$$

where:

Conc_C = concentration of component of interest,
A_C = peak area of component,
$(A \times R_F)_{\mathrm{ALL}}$ = sum of peak areas times their respective response factors relative to a standard, and
R_{F_C} = response factor of component to the same standard.

15. Recommended Form for Writing Gas Chromatographic Methods

15.1 *General*—Not all of the steps outlined in this section may be needed to describe adequately a method. A number of variations in procedure format are shown in the publication, ASTM Standards in Chromatography.[7] Ideally, the procedure should be written so that it can be followed by a person with the equivalent of a high school-chemistry understanding or six to twelve months of practical laboratory experience. Critical steps should be identified along with any reasons that show why this step is necessary to achieve a successful analysis. Any involved procedures should be written in an Appendix so that the main points in the procedure can be read more easily.

15.2 *Recommended Form:*

15.2.1 *Title*—The title should be concise, but complete

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[7] *ASTM Standards on Chromatography*, ASTM, 1981.

enough to identify the component(s) analyzed, the nature of the method (gas chromatography), the detector, and the materials to which it is applicable. Select words that easily lend themselves to indexing.

15.2.2 *Scope*—State as clearly as possible the range of application of method. In a separate paragraph, note interferring substances or any significant limitations of the method. This material could be placed in a later section (15.2.5), if an involved description is necessary.

15.2.3 *Pertinent Documents or References:*

15.2.3.1 *ASTM Standards.*

15.2.3.2 *Other Standard Methods*—Include any standard methods.

15.2.4 *Summary of the Method*—Describe the method in a general way, without going into details of the procedure. It may be appropriate to touch briefly on the following points: sample introduction technique, column dimensions and type of tubing material, nature of the packing material, mesh size of support or adsorbent, liquid phase loading (if a liquid phase was used), isothermal or programmed temperature and detector type (thermal conductivity, flame ionization, electron capture, *etc.*).

15.2.5 *Significance and Application*—Use this section for a more detailed discussion than can be fitted in the Scope.

15.2.6 *Definitions*—Include special definitions in this section. General chromatographic definitions are already available in Practice E 355, to which reference can be made.

15.2.7 *Interferences*—Use this section for a more detailed discussion than can be fitted into the Scope.

15.2.8 *Special Comments*—Use this section to include a description of special requirements needed to achieve a successful analysis.

15.2.9 *Safety Precautions*—If the method involves hazards, insert a warning to this effect. Point out the nature of the hazards, and describe precautionary measures which must be taken. Refer to the latest OSHA regulations regarding all materials used in this procedure.

15.2.10 *Gas Chromatographic System*—List and describe the apparatus. Describe the essential features of the apparatus that are necessary to achieve the desired analysis. Avoid the use of trade names. Include schematic drawings or photographs if they are needed to clarify or supplement the text. The gas chromatographic conditions can be either summarized in a table, as in Table 6, or in the text as follows:

15.2.10.1 *Sample Injection Port*—Construction: stainless steel, glass liner, fitted for on-column injection with a glass or metal column, etc. Temperature at which used.

15.2.10.2 *Column Oven*—Isothermal or temperature programmed operation: give temperatures and programming rates required.

15.2.10.3 *Detector*—Type (flame ionization, thermal conductivity, etc.), temperature of operation, sensitivity required. Detector gases used and flow rates.

15.2.10.4 *Recorder*—Operating range (in millivolts), chart speed, time for full-scale deflection of pen.

15.2.10.5 *Integrator*—Note operating characteristics of integrator and parameters used.

15.2.11 *Preparation and Installation of the Chromatographic Column:*

15.2.11.1 *Tubing Material*—Note the type of material, as stainless steel, nickel, glass, or glass-lined tubing, as well as the dimensions (outer diameter and inner diameter, or wall thickness and length). Any pretreatment of the column material, solvent washing, or silanization should be mentioned.

15.2.11.2 *Partitioning Phase*—Solid adsorbent, if used (type and mesh size). Coated support if used (liquid phase, percent loading and coating procedure, support type and pretreatment, and mesh size). Note sources for special materials in footnotes. Provide preparation and purification method for materials not commercially available. In an Appendix, note other liquid phases that have been successfully used in this analysis.

15.2.11.3 *Column Preparation*—Describe the procedure used to pack the column. Note the amount of packing in the column.

15.2.11.4 *Column Installation*—Note if the column is set up for back-flushing, if a sample fraction is removed on a pre-column, or other special column arrangement.

15.2.11.5 *Column Conditioning*—Provide the column conditioning procedure.

15.2.11.6 *Column Evaluation*—Give the procedure for evaluating the column. Calculation of the resolution between two components in a standard mixture will often be sufficient. Provide some estimate of column life and signs of degrading column performance (loss of resolution, peak broadening, or tailing). Provide examples of good and bad chromatograms.

15.2.12 *General Apparatus*—Volumetric flasks and pipets, microsyringes for sample introduction, balance (capacity and sensitivity), heat lamps, hot plates, etc.

15.2.13 *Reagents and Materials:*

15.2.13.1 *Chemicals and Reagents*—Include derivatizing reagents. Note purity, or purification methods, if required.

15.2.13.2 *Calibration Standards*—Note purity required.

15.2.13.3 *Gas (or Gases)*—Carrier gas, fuel gases for flame ionization detector, special gas for electron capture detector, etc. Note purity required.

15.2.14 *Calibration*—Describe in detail the calibration procedure. State whether pure components or standard mixtures are used and the basis of measurement. Include equations and describe the preparation of any calibration charts. Show the calibration curve. If a trace method is described, provide a chromatogram of the lowest detectable amount. Lengthy procedures, such as the development of complex equations, or the preparation of standard mixtures, should be placed in a section of an Appendix.

15.2.15 *Procedure*—Include, in proper sequence, directions for carrying out the method. Refer to the pertinent parts of the calibration procedure in 15.2.14. Do not repeat these steps here. Possible subheadings are as follows:

15.2.15.1 *Final Conditioning and Adjustment of the Gas Chromatographic System*—This section is intended to include adjustment and verification of the state of the chromatographic system before analytical use.

15.2.15.2 *Sampling*—Careful attention must be given to the sampling techniques since representative samples are essential to achieve successful analysis. Include special directions that may be required for taking samples, for preservation of samples, and for special treatment of samples prior to injection.

TABLE 6 Summary of Gas Chromatographic Conditions

Column	Length, outside diameter, and internal diameter (or outside diameter and wall thickness); weight percent of specified stationary phase; specified solid support (mesh size and treatment; for example, acid-washed, silanized); approximate amount of total column packing in a given column length.
Temperatures:	
Sample inlet system	°C
Detector	°C
Column	
If isothermal	°C
If programmed	
Initial	°C (note any hold time at initial temperature)
Final	°C
Heating rate	°C/min
(State specific temperatures and times if isothermal operation and temperature programming are combined)	
Carrier gas	(specify)
Flow rate	cm^3/min^A
Detector	TCD, FID, ECD
Detector Sensitivity	Flame ionization detector—amps full scale deflection, AFS^B Detector voltage or bridge current, if applicable.
Recorder characteristics	mV range, speed of full-scale deflection
Chart speed	cm (or in.)/min
Sample size	µL (or cm^3, if gas)

[A] Flow rate is best measured at the column or detector outlet, at the analytical temperature and flow rate.

[B] A flame ionization detector is assumed to be operated under optimum hydrogen and air flow rates, unless otherwise specified.

15.2.15.3 The remainder of the steps leading to the chromatogram.

15.2.15.4 *Typical Chromatogram*—Show, in a figure, a plot of the retention time (in minutes) versus the detector response. Label the known peaks (including the dead volume or unadsorbed gas peak) and indicate in parentheses the attenuation for each peak.

NOTE 8—When determining the retention time of the unadsorbed peak, the retention time of air is used for thermal conductivity detectors, methane for flame ionization detectors, and methylene chloride lead space vapors for ECDs.

15.2.15.5 *Retention Time Data*—Include a table listing retention times and relative retention times for all compounds of interest, for all recommended columns. Identify the unadsorbed peak and the reference material used for relative retention time calculations.

15.2.16 *Calculation*—State the reference point on which the calculations are based (for example, sample as received), the terms in which the results are finally obtained (weight, volume, or mole percent), and whether or not these values are normalized. Present the calculations in equation form, using letter symbols to designate variable values and numerical values of constants. Define the letter symbols in a legend immediately following the calculation equation.

15.2.17 *Report*—Show limits to be reported.

15.2.18 *Precision*—Limiting values for precision should be based on cooperative test results. Judgment as to the acceptability of results (95 % probability) should be based on the following criteria.

15.2.18.1 *Repeatability*—The following wording should be used: Duplicate results by the same operator should not be considered suspect unless they differ by more than the following amounts: (insert determined limits in tabular form).

15.2.18.2 *Reproducibility*—The following wording should be used: The result submitted by each of two laboratories should not be considered suspect unless the two results differ by more than the following amounts: (insert determined amounts in tabular form).

15.2.19 *Appendixes*—Supplementary information may be included in one or more Appendixes to the report. Examples of such information are: technique to improve column life, directions to clean the apparatus, leak check procedures, procedures to optimize column performance, development of equations used in the calculations, and precautions to avoid common causes of errors, etc.

16. Keywords

16.1 gas chromatography; GC; packed columns

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

* * *

Document Name: ASTM E283: Standard Test Method for Determining the Rate of Air Leakage Through Exterior Windows, Curtain Walls, and Doors

CFR Section(s): 10 CFR 434.402.2

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 283 – 91 (Reapproved 1999)

An American National Standard

Standard Test Method for Determining Rate of Air Leakage Through Exterior Windows, Curtain Walls, and Doors Under Specified Pressure Differences Across the Specimen

This standard is issued under the fixed designation E 283; 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 test method provides a standard laboratory procedure for determining the air leakage rates of exterior windows, curtain walls, and doors under specified differential pressure conditions across the specimen. The test method described is for tests with constant temperature and humidity across the specimen.[1]

1.2 This laboratory procedure is applicable to exterior windows, curtain walls, and doors and is intended to measure only such leakage associated with the assembly and not the installation. The test method can be adapted for the latter purpose.

Note 1—Performing tests at non-ambient conditions or with a temperature differential across the specimen may affect the air leakage rate. This is not addressed by this test method.

1.3 This test method is intended for laboratory use. Persons interested in performing field air leakage tests on installed units should reference Method E 783.

1.4 Persons using this procedure should be knowledgeable in the areas of fluid mechanics, instrumentation practices, and shall have a general understanding of fenestration products and components.

1.5 Throughout this test method, SI units are listed first in accordance with E-6 metric policy, and shall be considered the primary units. Non-SI units are provided in parenthesis.

1.6 *This standard does not purport to address all of the safety problems, 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 hazard statement see Section 7.

2. Referenced Documents

2.1 *ASTM Standards:*

E 631 Terminology of Building Constructions[2]

E 783 Test Method for Field Measurement of Air Leakage Through Installed Exterior Windows and Doors[2]

3. Terminology

3.1 *Definitions*—Terms used in this standard are defined in Terminology E 631.

3.2 *Descriptions of Terms Specific to This Standard:*

3.2.1 *air leakage rate* (q_A or q_L), $m^3/s{\cdot}m^2$ ($ft^3/min{\cdot}ft^2$), or $m^3/s{\cdot}m$ ($ft^3/min{\cdot}ft$)—the air leakage per unit of specimen area (*A*) or per unit length of operable crack perimeter (*L*).

3.2.2 *extraneous air leakage* (Q_e), m^3/s (ft^3/min)—the volume of air flowing per unit of time through the test chamber and test apparatus, exclusive of the air flowing through the test specimen, under a test pressure difference and test temperature difference, converted to standard conditions.

3.2.2.1 *Discussion*—Extraneous leakage is the sum of all leakage other than that intended to be measured by the test.

3.2.3 *specimen*—the entire assembled unit submitted for test as described in Section 7.

3.2.4 *specimen air leakage* (Q_s), m^3/s (ft^3/min)—the volume of air flowing per unit of time through the specimen under a test pressure difference and test temperature difference, converted to standard conditions.

3.2.5 *specimen area* (*A*), m^2 (ft^2)—the area determined by the overall dimensions of the frame that fits into the rough opening.

3.2.6 *standard test conditions*—in this test method, dry air at:

Pressure—101.3 kPa (29.92 in. Hg)
Temperature—20.8°C (69.4°F)
Air Density—1.202 kg/m^3 (0.075 lbm/ft^3)

3.2.7 *test pressure differences, Pa* (lbf/ft^2)—the specified differential static air pressure across the specimen.

3.2.8 *total air flow* (Q_t), m^3/s (ft^3/min)—the volume of air flowing per unit of time through the test chamber and test apparatus, inclusive of the air flowing through the test specimen, under a test pressure difference and test temperature difference, converted to standard conditions.

3.2.9 *unit length of operable crack perimeter* (*L*), m (ft)—the sum of all perimeters of operable ventilators, sash, or doors contained in the test specimen, based on the overall dimensions of such parts. Where two such operable parts meet the two adjacent lengths of perimeter shall be counted as only one length.

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[1] This test method is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.51 on Component Performance of Windows, Curtain Walls, and Doors.

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[2] *Annual Book of ASTM Standards*, Vol 04.11.

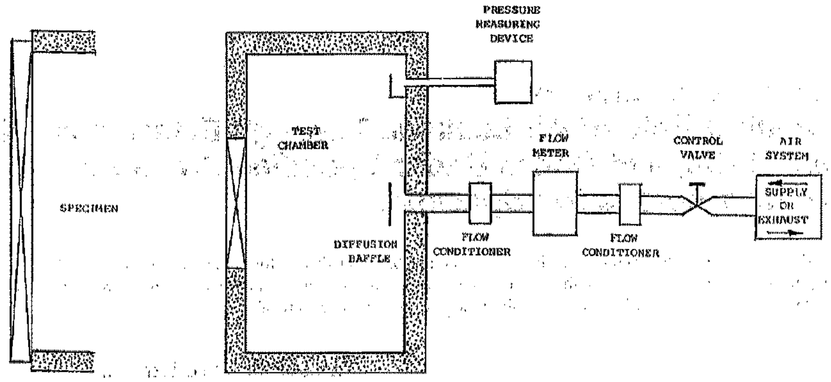

FIG. 1 General Arrangement of the Air Leakage Apparatus

4. Summary of Test Method

4.1 The test consists of sealing a test specimen into or against one face of an air chamber, supplying air to or exhausting air from the chamber at the rate required to maintain the specified test pressure difference across the specimen, and measuring the resultant air flow through the specimen.

5. Significance and Use

5.1 This test method is a standard procedure for determining the air leakage characteristics under specified air pressure differences at ambient conditions.

NOTE 2—The air pressure differences acting across a building envelope vary greatly. The factors affecting air pressure differences and the implications or the resulting air leakage relative to the environment within buildings are discussed in the literature.[3,4,5] These factors should be fully considered in specifying the test pressure differences to be used.

5.2 Rates of air leakage are sometimes used for comparison purposes. Such comparisons may not be valid unless the components being tested and compared are of essentially the same size, configuration, and design.

6. Apparatus

6.1 The description of the apparatus in this section is general in nature. Any suitable arrangement of equipment capable of maintaining the required test tolerances is permitted.

6.2 *Test Chamber*—A well sealed box, wall, or other apparatus into or against which the specimen is mounted and secured for testing. An air supply shall be provided to allow a positive or negative pressure differential to be applied across the specimen without significant extraneous losses. The chamber shall be capable of withstanding the differential test pressures that may be encountered in this procedure. At least one static air pressure tap shall be provided on each side of the specimen to measure the test pressure differences. The pressure tap shall be located in an area of the chamber in which pressure readings will not be affected by any supply air. The air supply opening to the chamber shall be located in an area in which it does not directly impinge upon the test specimen.

6.2.1 *Supply Air System*—A controllable blower, exhaust fan, or reversible blower designed to provide the required air flow at the specified test pressure difference. The system should provide essentially constant air flow at the specified test pressure difference for a time period sufficient to obtain readings of air flow.

6.2.2 *Pressure Measuring Apparatus*—A device to measure the differential test pressures to ± 2 % of setpoint or ± 2.5 Pa (± 0.01 in. of water column), whichever is greater.

6.2.3 *Air Flow Metering System*—A device to measure the air flow into the test chamber or through the test specimen. The air flow measurement error shall not exceed ± 5 % when the air flow equals or exceeds 9.44×10^{-4} m^3/s (2 ft^3/min) or ± 10 % when the air flow is less than $9.44^{-4} \times 10$ m^3/s (ft^3/min).

NOTE 3—At lower flows a greater percentage of errors will be acceptable. If higher precision is required, special flow metering techniques are necessary. The accuracy of the specimen air leakage flow measurement is affected by the accuracy of the flowmeter and the amount of extraneous air leakage. (See Annex A1.)

7. Hazards

7.1 **Precaution**—Glass breakage may occur at the test pressure differences applied in this test. Adequate precautions should be taken to protect personnel.

8. Test Specimen

8.1 The test specimen for a wall shall be of sufficient size to determine the performance of all typical parts of the wall system. For curtain walls or walls constructed with prefabricated units, the specimen width shall be not less than two typical units plus the connections and supporting elements at both sides, and sufficient to provide full loading on at least one typical vertical joint or framing member, or both. The height shall be not less than the full building story height or the height of the unit, whichever is greater, and shall include at least on full horizontal joint, accommodating vertical expansion, such joint being at or near the bottom of the specimen, as well as all connections at top and bottom of the units.

8.1.1 All parts of the wall test specimen shall be full size

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[3] Available from American Society of Heating, Refrigeration, and Air-Conditioning Engineers, 1791 Tullie Circle N.E., Atlanta, GA 30329. ASHRAE Handbook of Fundamentals, 1989.

[4] Fluid Meters—Their Theory and Application, 5th Edition, 1959.

[5] Available from American Society of Mechanical Engineers, 345 E. 47th St., New York, NY 10017. Power Test Code, 2nd Edition, 1956, Part 5, Chapter 4, "Flow Measurements."

using the same materials, details, and methods of construction and anchorage as used on the actual building.

8.1.2 Conditions of structural support shall be simulated as accurately as possible.

8.2 The test specimen for a window, door, or other component shall consist of the entire assembled unit, including frame and anchorage as supplied by the manufacturer for installation in the building. If only one specimen is to be tested the selection shall be determined by the specifying authority.

NOTE 4—The air leakage rate is likely to be a function of size and geometry of the specimen.

9. Calibration

9.1 Specific procedures for calibration of the total air flow measurement system are being developed in a separate ASTM document. When complete, that document will be referenced. However, all test apparatus shall be calibrated at a minimum of every 6 months to the tolerances established in Section 6. The procedures for this calibration are, at this time, the responsibility of the testing agency. Calibration should be conducted at or near the environmental conditions (temperature, relative humidity, and so forth) under which the tests are to be conducted and to which the test apparatus is to be exposed.

10. Test Conditions

10.1 The specifying authority shall supply the following information:

10.1.1 Specimen test size,

10.1.2 Test pressure difference (if no value is designated, 75 Pa (1.57 lb/ft^2)), and

10.1.3 Direction of air flow, exfiltration or infiltration. (If none is specified, the test shall be infiltration.)

10.2 *Air Leakage Rate*—Basis for reporting air leakage rate shall be total air leakage m^3/h (ft^3/min), per unit length of operable crack perimeter, m^3/h – m (ft^3/min·ft), and per unit area of outside frame dimension, m^3/h – m^2 (ft^3/min·ft ^{2}).

11. Procedure

11.1 Remove any sealing material or construction that is not normally a part of the assembly as installed in or on a building. Fit the specimen into or against the chamber opening. Installation should be such that no parts or openings of the specimen are obstructed.

NOTE 5—Nonhardening mastic compounds or pressure sensitive tape can be used effectively to seal the test specimen to the chamber, and to achieve air tightness in the construction of the chamber. These materials can also be used to seal a separate mounting panel to the chamber. Rubber gaskets with clamping devices may also be used for this purpose, provided that the gasket is highly flexible and has a narrow contact edge.

11.2 Without disturbing the seal between the specimen and the test chamber, adjust all hardware, ventilators, balances, sash, doors, and other components included as an integral part of the specimen so that their operation conforms to test method requirements.

11.3 To ensure proper alignment and weather seal compression, fully open, close, and lock each ventilator, sash, or door five times prior to testing.

11.4 Adjust the air flow through the test chamber to provide the specified test pressure difference across the test specimen. When the test conditions are stabilized, record the air flow through the flowmeter and the test pressure difference. This measured air flow is designated the total air flow, Q_t. Measure the barometric pressure, and temperature of the air at the test specimen.

11.5 Eliminate extraneous chamber leakage, or, if this is impractical, measure the amount of such leakage with the specimen sealed, at the air pressure differences to be exerted during the air leakage tests. Designate this measured air flow as the extraneous air flow, Q_e.

12. Calculation

12.1 Express the total air flow, Q_t, and the extraneous leakage, Q_e, in terms of flow at standard conditions, Q_{st}, using the Eq 1 and 2.

$$Q_{st} = Q(W/W_s)^{1/2} \tag{1}$$

$$W = 3.485 \times 10^{-3} \, (B/(T + 273)) \tag{2}$$

where:

Q = airflow at non-standard conditions,
Q_{st} = airflow corrected to standard conditions,
W_s = density of air at reference standard conditions—1.202 kg/m^3 (0.075 lb/ft^3),
W = density of air at the test site, kg/m^3(lb/ft^3),
B = barometric pressure at test site corrected for temperature, Pa (in. Hg), and
T = temperature of air at flowmeter, °C (°F).

NOTE 6—Use the equation $W = 1.326 \, (B/(T + 460))$ for calculating in inch-pound units.

12.2 Express the air leakage through the test specimen as follows:

$$Q_s = Q_t - Q_e \tag{3}$$

where:

Q_s = air leakage through the test specimen, m^3/s (ft^3/min), at standard conditions.

12.3 Calculate the rate of air leakage for the test specimen according to 12.3.1 and 12.3.2.

12.3.1 To calculate q_L rate of air leakage per unit of length of operable crack perimeter use Eq 4:

$$q_L = Q_s/L, \ \text{m}^3/\text{h} \cdot \text{m} \ (\text{ft}^3/\text{min} \cdot \text{ft}) \tag{4}$$

12.3.2 To calculate q_A rate of air leakage per unit area:

$$q_A = Q_s/A, \ \text{m}^3/\text{h} \cdot \text{m}^2 \ (\text{ft}^3/\text{min} \cdot \text{ft}^2) \tag{5}$$

13. Report

13.1 Report the following information:

13.1.1 *General*—Testing agency, date and time of test, and date of report.

13.1.2 *Sample Description*—Manufacturer, model, operation type, materials, and other pertinent information; description of the locking and operating mechanisms if applicable; glass thickness, type and method of glazing; weather seal dimensions, type, and material; and crack perimeter and specimen area.

13.1.3 *Drawings of Specimen*—Detailed drawings of the specimen showing dimensioned section profiles, sash or door dimensions and arrangement, framing location, panel arrangement, installation and spacing of anchorage, weatherstripping, locking arrangement, hardware, sealants, glazing details, and any other pertinent construction details. Any modifications made on the specimen to obtain the reported test values shall be noted.

13.1.4 *Test Parameters*—List or describe the specified test pressure difference(s), whether the tests were conducted for infiltration or exfiltration, and whether a positive or negative test pressure was used.

13.1.5 *Pressure Differences and Leakage*— A statement or tabulation of the pressure differentials exerted across the specimen during the test and the corresponding specimen air leakage (Q_s) and the two air leakage rates (q_L and q_A).

13.1.6 *Compliance Statement*—A statement that the tests were conducted in accordance with this test method, or a complete description of any deviation from this test method. When the tests are conducted to check for conformity of the specimen to a particular performance specification, the specification shall be identified.

13.2 If several identical specimens are tested, the results for each specimen shall be reported, each specimen being properly identified, particularly with respect to distinguishing features or differing adjustment. A separate drawing for each specimen shall not be required if all differences between the specimens are noted on the drawings provided.

14. Precision and Bias

14.1 The precision and bias of this test method has not been determined.

15. Keywords

15.1 air leakage; curtain walls; doors; fenestration; laboratory method; static pressure chamber; windows

ANNEX

(Mandatory Information)

A1. ERRORS IN WINDOW AIR LEAKAGE MEASUREMENT

A1.1 Terminology

A1.1.1 *Symbols*:

A1.1.1.1 Q_s = air flow through specimen.

A1.1.1.2 Q_{ts} = total air flow.

A1.1.1.3 Q_{es} = extraneous air flow.

A1.1.1.4 Δ = delta.

NOTE A1.1—Symbols A1.1.1.1-A1.1.1.4 have been converted to standard conditions.

A1.2 In the apparatus using a supply air system, $Q_s = Q_{ts} - Q_{es}$, the extraneous air leakage (Q_{es}) represents all the air leakage leaving the chamber which does not pass through the specimen proper. This includes leakage passing through the chamber walls and around the specimen mounting. When the mounting panel is used, leakage between the chamber and the panel contributes to extraneous leakage. The extraneous leakage flow is a function of the pressure difference between the chamber and the room, which is also the test specimen difference.

A1.3 The total error in the specimen flow determination (neglecting errors in the air density determination) is as follows:

$$\Delta Q_s/Q_s = [\Delta Q_{ts}/(Q_{ts} \cdot Q_{es})] \pm [\Delta Q_{es}/(Q_{ts} \cdot Q_{es})] \qquad (A1.1)$$

A1.3.1 According to 6.2.3, the air flow through the test specimen is to be determined with an error no greater than $\Delta Q_s/Q_s = \pm 5\ \%$ if the extraneous leakage is accurate to $\Delta Q_{es}/Q_{es} = \pm 10\ \%$ and Q_{es} is 10 % of Q_s, then the contribution of the extraneous leakage to the overall error in Eq 2 is ± 1 %. (Note that the error attributed to the extraneous leakage determination is a function not only of the accuracy of the flow meter used in the determination, but also of the constancy of the leakage from the time of determination to the time of test.) The error contributed by the flow meter to the total error is then limited to 4 %, but because $Q_{ts} = Q_s + Q_{es} = 1.10\ Q_s$ the accuracy required of the flowmeter is:

$$\Delta\left(\frac{Q_{ts}}{Q_t}\right) = \frac{4}{1.1}\ \% = 3.6\ \% \qquad (A1.2)$$

A1.3.2 It is seen that the major factor affecting the accuracy required of the flowmeter is the proportion of Q_{es} to Q_s. If $\Delta Q_{es}/Q_{es}$ remains at ±10 %, but Q_{ts} is 50 % of Q_s, the error contributed by the extraneous leakage becomes 5 % and no error can be tolerated in the flowmeter if the conditions of 6.2.5 are to be met—with Q_{es} in excess of 50 % it is impossible to achieve the required overall limit of error. Likewise, if the extraneous leakage is eliminated, the flowmeter error can be as great as 5 %.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

◆

Document Name: ASTM E29: Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

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Standards Body: American Society for Testing and Materials

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WASHINGTON, D.C.

Designation: E 29 – 90

Standard Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications[1]

This standard is issued under the fixed designation E 29; 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.

This standard has been approved for use by agencies of the Department of Defense. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This practice is intended to assist the various technical committees in the use of uniform methods of indicating the number of digits which are to be considered significant in specification limits, for example, specified maximum values and specified minimum values. Its aim is to outline methods which should aid in clarifying the intended meaning of specification limits with which observed values or calculated test results are compared in determining conformance with specifications. Reference to this practice is valid only when a choice of method has been indicated, that is, either *absolute method or rounding-off method.*

1.2 This practice is intended to be used in determining conformance with specifications when the applicable ASTM specifications or standards make direct reference to this practice.

1.3 This practice describes two commonly accepted methods of rounding data, identified as the Absolute Method and the Rounding-Off Method. In the application of this practice to a specific material or materials it is essential to specify which method is intended to apply. In the absence of such specification, reference to this practice, which expresses no preference as to which method should apply, would be meaningless. The choice of method is arbitrary, depending upon the current practice of the particular branch of industry or technology concerned, and should therefore be specified in the prime publication.

1.4 Section 7 of this practice gives guidelines for use in recording, calculating, and reporting the final result for test data.

2. Referenced Documents

2.1 *ASTM Standards:*

E 456 Terminology Related to Quality and Statistics[2]

E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System)[2]

2.1 *ANSI Standard:*

ANSI Z25.1 Rules for Rounding Off Numerical Values[3]

3. Terminology

3.1 *significant digit, n*—any of the figures 0 through 9, excepting leading zeros and some trailing zeros, which is used with its place value to denote a numerical quantity to some desired approximation.

3.1.1 The digit zero may either indicate a specific value or indicate place only. Zeros leading the first nonzero digit of a number indicate order of magnitude only and are not significant digits. For example, the number 0.0034 has two significant digits. Zeros trailing the last nonzero digit for numbers represented with a decimal point are significant digits. For example, the numbers 1270. and 32.00 each have four significant digits. The significance of trailing zeros for numbers represented without use of a decimal point can only be identified from knowledge of the source of the value. For example, a tensile strength, stated as 140 000 psi, may have as few as two or as many as six significant figures. To avoid ambiguity, the exponential notation may be used; thus, 1.40×10^5 psi indicates that the tensile strength is reported to the nearest 0.01×10^5 or 1000 psi.

4. Expression of Numerical Requirements

4.1 The unqualified statement of a numerical limit, such as "2.50 in. max," cannot, in view of different established practices and customs, be regarded as carrying a definite operational meaning concerning the number of digits to be retained in an observed or a calculated value for purposes of determining conformance with specifications.

4.2 *Absolute Method*—In some fields, specification limits of 2.5 in. max, 2.50 in. max, and 2.500 in. max are all taken to imply the same absolute limit of exactly two and a half inches and for purposes of determining conformance with specifications, an observed value or a calculated value is to be compared directly with the specified limit. Thus, any deviation, however small, outside the specification limit signifies nonconformance with the specifications. This will be referred to as the *absolute method.*

4.3 *Rounding-Off Method*—In other fields, specification limits of 2.5 in. max, 2.50 in. max, 2.500 in. max are taken to imply that, for the purposes of determining conformance with specifications, an observed value or a calculated value should be rounded off to the nearest 0.1 in., 0.01 in., 0.001 in., respectively, and then compared with the specification limit. This will be referred to as the *rounding-off method.*

5. Absolute Method

5.1 *Where Applicable*—The absolute method applies

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[1] This practice is under the jurisdiction of ASTM Committee E-11 on Quality and Statistics and is the direct responsibility of Subcommittee E11.03 on Statistical Analysis and Control Techniques.

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

[3] Available from American National Standards Institute, 1430 Broadway, New York, NY 10018.

where it is the intent that all digits in an observed value or a calculated value are to be considered significant for purposes of determining conformance with specifications. Under these conditions, the specified limits are referred to as absolute limits.

5.2 *How Applied*—With the absolute method, an observed value or a calculated value is not to be rounded off, but is to be compared directly with the specified limiting value. Conformance or nonconformance with the specification is based on this comparison.

5.3 *How Expressed*—This intent may be expressed in the standard in one of the following forms:

5.3.1 If the absolute method is to apply to all specified limits in the standard, this may be indicated by including the following sentence in the standard:

> For purposes of determining conformance with these specifications, all specified limits in this standard are absolute limits, as defined in ASTM Practice E 29, for Using Significant Digits in Test Data to Determine Conformance with Specifications.

5.3.2 If the absolute method is to apply to all specified limits of some general type in the standard (such as dimensional tolerance limits), this may be indicated by including the following sentence in the standard:

> For purposes of determining conformance with these specifications, all specified (dimensional tolerance) limits are absolute limits, as defined in ASTM Practice E 29, Using Significant Digits in Test Data to Determine Conformance with Specifications.

5.3.3 If the absolute method is to apply to all specified limits given in a table, this may be indicated by including a footnote with the table as follows:

Width, in.	Width Tolerances[A], Plus and Minus, in. 0.032 in. and under in Thickness	 Over 0.032 in. in Thickness
2 and under	0.005	0.010
Over 2 to 8 incl	0.008	0.013
Over 8 to 14 incl	0.010	0.015
Over 14 to 20 incl	0.013	0.018

[A] Tolerance limits specified are absolute limits as defined in ASTM Practice E 29, for Using Significant Digits in Test Data to Determine Conformance with Specifications.

6. Rounding-Off Method

6.1 *Where Applicable*—The rounding-off method applies where it is the intent that a limited number of digits in an observed value or a calculated value are to be considered significant for purposes of determining conformance with specifications.

6.2 *How Applied*—With the rounding-off method, an observed value or a calculated value should be rounded off by the procedure prescribed in 4.3 to the nearest unit in the designated place of figures stated in the standard, as, for example, "to the nearest 100 psi," "to the nearest 10 ohms," "to the nearest 0.1 percent," etc. The rounded-off value should then be compared with the specified limit, and conformance or nonconformance with the specification based on this comparison.

6.3 *How Expressed*—This intent may be expressed in the standard in one of the following forms:

6.3.1 If the rounding-off method is to apply to all specified limits in the standard, and if all digits expressed in the specification limit are to be considered significant, this may be indicated by including the following statement in the standard:

> The following applies to all specified limits in this standard: For purposes of determining conformance with these specifications, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of ASTM Practice E 29, for Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.3.2 If the rounding-off method is to apply only to the specified limits for certain selected requirements, this may be indicated by including the following statement in the standard:

> The following applies to specified limits for requirements on (tensile strength), (elongation), and (. . .) given in . . ., (applicable section number and title) and (. . .) of this standard: For purposes of determining conformance with these specifications, an observed value or a calculated value shall be rounded off to the nearest (1000 psi) for (tensile strength), to the nearest (1 percent) for (elongation), and to the nearest (. . .) for (. . .) in accordance with the rounding-off method of ASTM Practice E 29 Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.3.3 If the rounding-off method is to apply to all specified limits in a table, this may be indicated by a note in the manner shown in the following examples:

6.3.3.1 *Example 1*—Same significant digits for all items:

	Chemical Composition, %
Copper	4.5 ± 0.5
Iron	1.0 max
Silicon	2.5 ± 0.5
Other constituents (magnesium + zinc + manganese)	0.5 max
Aluminum	remainder

NOTE 1—For purposes of determining conformance with these specifications, an observed value or a calculated value shall be rounded off to the nearest 0.1 percent, in accordance with the rounding-off method of ASTM Practice E 29, for Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.3.3.2 *Example 2*—Significant digits not the same for all items; similar requirements:

	Chemical Composition, percent min	 max
Nickel	57	
Chromium	14	18
Manganese		3
Silicon	. . .	0.40
Carbon	. . .	0.25
Sulfur	. . .	0.03
Iron	remainder	

NOTE 2—For purposes of determining conformance with these specifications, an observed value or a calculated value shall be rounded off "to the nearest unit" in the last right-hand significant digit used in expressing the limiting value, in accordance with the round-off method of ASTM Practice E 29, Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.3.3.3 *Example 3*—Significant digits not the same for all items; dissimilar requirements:

	Tensile Requirements
Tensile strength, psi	60 000 to 72 000
Yield point, min, psi	33 000
Elongation in 2 in., min %	22

NOTE 3—For purposes of determination of conformance with these specifications, an observed value or a calculated value shall be rounded off to the nearest 1000 psi for tensile strength and yield point and to the nearest 1 percent for elongation, in accordance with the rounding-off

method of ASTM Practice E 29 for Using Significant Digits in Test Data to Determine Conformance with Specifications.

6.4 *Rounding-Off Procedure*—The actual rounding-off procedure[4] shall be as follows:

6.4.1 When the digit next beyond the last place to be retained is less than 5, retain unchanged the digit in the last place retained.

6.4.2 When the digit next beyond the last place to be retained is greater than 5, increase by 1 the digit in the last place retained.

6.4.3 When the digit next beyond the last place to be retained is 5, and there are no digits beyond this 5, or only zeros, increase by 1 the digit in the last place retained if it is odd, leave the digit unchanged if it is even. Increase by 1 the digit in the last place retained, if there are digits beyond this 5.

6.4.4 This rounding-off procedure may be restated simply as follows: When rounding off a number to one having a specified number of significant digits, choose that which is nearest. If two choices are possible, as when the digits dropped are exactly a 5 or a 5 followed only by zeros, choose that ending in an even digit. Table 1 gives examples of applying this rounding-off procedure.

TABLE 1 Examples[A] of Rounding Off

Specified Limit	Observed Value or Calculated Value	To Be Rounded Off to Nearest	Rounded-Off Value to be Used for Purposes of Determining Conformance	Conforms with Specified Limit
Yield point, 36 000 psi, min	35 940	100 psi	35 900	no
	35 950	100 psi	36 000	yes
	35 960	100 psi	36 000	yes
Nickel, 57 %, min	56.4	1 %	56	no
	56.5	1 %	56	no
	56.6	1 %	57	yes
Water extract conductivity, 40 micromhos/cm, max	40.4	1 micromho/cm	40	yes
	40.5	1 micromho/cm	40	yes
	40.6	1 micromho/cm	41	no
Sodium bicarbonate 0.5 %, max	0.54	0.1 %	0.5	yes
	0.55	0.1 %	0.6	no
	0.56	0.1 %	0.6	no

[A] These examples are meant to illustrate rounding rules and do not necessarily reflect the usual number of digits associated with these test methods.

6.5 The rounded-off value should be obtained in one step by direct rounding off of the most precise value available and not in two or more successive roundings. For example: 89 490 psi rounded off to the nearest 1 000 psi is at once 89 000; it would be incorrect to round off first to the nearest 100, giving 89 500 and then to the nearest 1 000, giving 90 000.

6.6 *Special Case, Rounding Off to the Nearest 50, 5, 0.5, 0.05, etc.*—If in special cases it is desired to specify rounding off to the nearest 50, 5, 0.5, 0.05, etc., this may be done by so indicating in the standard. In order to round off to the nearest 50, 5, 0.5, 0.05, etc., double the observed or calculated value, round off to the nearest 100, 10, 1.0, 0.10, etc., in accordance with the procedure in 6.4, and divide by 2. For example, in rounding off 6 025 to the nearest 50, 6 025 is doubled giving 12 050 which becomes 12 000 when rounded off to the nearest 100 (6.4.3). When 12 000 is divided by 2, the resulting number, 6 000, is the rounded-off value of 6 025. In rounding off 6 075 to the nearest 50, 6 075 is doubled giving 12 150 which becomes 12 200 when rounded off to the nearest 100 (6.4.3). When 12 200 is divided by 2, the resulting number, 6 100, is the rounded-off value of 6 075.

7. Guidelines for Retaining Significant Figures in Calculation and Reporting of Test Results

7.1 *General Discussion*—Rounding test results avoids a misleading impression of precision while preventing loss of information due to coarse resolution. Any approach to retention of significant digits of necessity involves some loss of information; therefore, the level of rounding should be carefully selected considering both planned and potential uses for the data. The number of significant digits must, first, be adequate for comparison against specification limits (see 6.2). The following guidelines are intended to preserve the data for statistical summaries. For certain purposes, such as where calculations involve differences of measurements close in magnitude, and for some statistical calculations, such as paired t-tests, autocorrelations, and nonparametric tests, reporting data to a greater number of significant digits may be advisable.

7.2 *Recording Test Data*—When recording direct measurements, as in reading marks on a buret, ruler, or dial, all digits known exactly, plus one digit which may be uncertain due to estimation, should be recorded. For example, if a buret is graduated in units of 0.1 mL, then an observation would be recorded as 9.76 mL where it is observed between 9.7 and 9.8 marks on the buret, and estimated about six tenths of the way between those marks. When the measuring device has a vernier scale, the last digit recorded is the one from the vernier.

7.2.1 The number of significant digits given by a digital display or printout from an instrument should be greater than or equal to those given by the rule for reporting test results in 7.4 below.

7.3 *Calculation of Test Result from Test Data*—When calculating a test result from test data, avoid rounding of intermediate quantities. As far as is practicable with the calculating device or form used, carry out calculations with the test data exactly and round only the final result.

7.4 *Reporting Test Results*—A suggested rule relates the significant digits of the test result to the precision of the measurement expressed as the standard deviation σ. The applicable standard deviation is the repeatability standard deviation (see Terminology E 456). Test results should be round to not greater than 0.5 σ nor less than 0.05 σ, provided that this value is not greater than the unit specified in the specification (see 6.2). When only an estimate, s, is available for σ, s may be used in place of σ in the preceding sentence.

Example: A test result is calculated as 1.45729. The standard deviation of the test method is estimated to be, 0.0052. Round to 1.457 or the nearest 0.001 since this rounding unit, 0.001, is between 0.05 σ = 0.00026 and 0.5 σ = 0.0026.

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[4] The rounding-off procedure given in this practice is the same as the one given in the American National Standard Rules for Rounding Off Numerical Values (ANSI Z25.1) and in the *ASTM Manual on Presentation of Data and Control Chart Analysis, STP 15-D.*

NOTE 4—A rationale for this rule is derived from representing the standard deviation of a rounded test result by $\sqrt{\sigma^2 + w^2/12}$ where σ is the standard deviation of the unrounded test result. The quantity $w/\sqrt{12}$ is the standard deviation of an error uniformly distributed over the range w. Rounding so that w is below 0.5 σ ensures that the standard deviation is increased by at most 1 %, while adding more digits would give a misleading impression of precision.

7.4.1 When no estimate of the standard deviation σ is known, then rules for retention of significant digits of computed quantities may be used to derive a number of significant digits to be reported, based on significant digits of test data.

7.4.1.1 The rule when adding or subtracting test data is that the result shall contain no significant digits beyond the place of the last significant digit of any datum.

Examples:

(*1*) 11.24 + 9.3 + 6.32 = 26.9, since the last significant digit of 9.3 is the first following the decimal place,

(*2*) 26.9 is obtained by rounding the exact sum, 26.86, to this place of digits.

(*3*) 926 − 923.4 = 3

140,000 + 91,460 = 231,000 when the first value was recorded to the nearest thousand.

7.4.1.2 The rule when multiplying or dividing is that the result shall contain no more significant digits than the value with the smaller number of significant digits.

Examples:

(*1*) 11.38 × 4.3 = 49, since the factor 4.3 has two siginificant digits

(*2*) (926 − 923.4)/4.3 = 0.6 Only one figure is significant since the numerator difference has only one significant digit.

7.4.1.3 The rules for logarithms and exponentials are: Digits of $ln(x)$ or $\log_{10}(x)$ are significant through the n-th place after the decimal when x has n significant digits. The number of significant digits of e^x or 10^x is equal to the place of the last significant digit in x after the decimal.

Examples: $ln(3.46) = 1.241$ to three places after the decimal, since 3.46 has three significant digits. $10^{3.46} = 2900$ has two significant digits, since 3.46 is given to two places after the decimal.

7.4.1.4 The rule for numbers representing exact counts or mathematical constants is that they are to be treated as having an infinite number of significant digits.

Examples:

(*1*) 1 − 0.23/2 = 0.88 where the numbers 1 and 2 are exact and 0.23 is an approximate quantity.

(*2*) A count of 50 pieces times a measured thickness 0.124 mm is 50 × 0.124 = 6.20 mm, having three significant figures.

(*3*) A measurement of 1.634 in. to the nearest thousandth, is converted to mm. The result, 1.634 × 25.4 = 41.50 mm, has four significant digits. The conversion constant, 25.4, is exact.

NOTE 5—More extensive discussion of dimensional conversion can be found in Practice E 380.

7.5 *Specification Limits*—When the rounding off method is to apply to given specified limits, it is desirable that the significant digits of the specified limits should conform to the precision of the test following the rule of 7.3. That is, the rounding unit for the specification limits should be between 0.05 and 0.5 times the standard deviation of the test.

7.6 *Averages and Standard Deviations*—When reporting the average and standard deviation of replicated measurements or repeated samplings of a material, a suggested rule for most cases is to round the standard deviation to two significant digits and round the average to the same last place of significant digits. When the number of observations is large (more than 15 when the lead digit of the standard deviation is 1, more than 50 with lead digit 2, more than 100 in other cases), an additional digit may be advisable.

7.6.1 Alternative approaches for averages include reporting $\bar{x}$ to within 0.05 to 0.5 times the standard deviation of the average $\sigma/\sqrt{n}$, or applying rules for retaining significant digits to the calculation of $\bar{x}$. The ASTM STP 15-D provides methods for reporting $\bar{x}$ and s for these applications.[4]

NOTE 6—A rationale for the suggested rule comes from the uncertainty of a calculated standard deviation s. The standard deviation of s based on sampling from a normal distribution with n observations is approximately $\sigma/\sqrt{2n}$. Reporting s to within 0.05 to 0.5 of this value, following the rule of 7.4, leads to two significant digits for most values of σ when the number of observations n is 100 or fewer.

Example: Analyses on six specimens give values of 3.56, 3.88, 3.95, 4.07, 4.21, and 4.47 for a constituent. The average and standard deviation, unrounded, are $\bar{x}$ = 4.0233. . . and s = 0.3089. . . . The suggested rule would report $\bar{x}$ and s as 4.02 and 0.31.

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Document Name: ASTM E408: Standard Test Methods for Total Normal Emittance of Surfaces Using Inspection-Meter Techniques

CFR Section(s): 16 CFR 460.5(b)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
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WASHINGTON, D.C.

Designation: E 408 – 71

Standard Methods of Test for
TOTAL NORMAL EMITTANCE OF SURFACES USING INSPECTION-METER TECHNIQUES[1]

This Standard is issued under the fixed designation E 408; 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.

1. Scope

1.1 These methods cover determination of the total normal emittance (Note 1) of surfaces by means of portable, inspection-meter instruments.

NOTE 1—Total normal emittance (ϵ_N) is defined as the ratio of the normal radiance of a specimen to that of a blackbody radiator at the same temperature. The equation relating ϵ_N to wavelength and spectral normal emittance [$\epsilon_N(\lambda)$] is

$$\epsilon_N = \int_0^\infty L_b(\lambda,T)\epsilon_N(\lambda)d\lambda / \int_0^\infty L_b(\lambda,T)d\lambda$$

where:

$L_b(\lambda,T)$ = Planck's blackbody radiation function = $c_1\pi^{-1}\lambda^{-5}(e^{c_2/\lambda T}-1)^{-1}$,
c_1 = 3.7415×10^{-16} W·m^2,
c_2 = 1.4388×10^{-2} m·K,
T = absolute temperature, K,
λ = wavelength, m,
$\int_0^\infty L_b(\lambda,T)d\lambda$ = $\Delta\pi^{-1}T^4$, and
Δ = Stefan-Boltzmann constant = 5.66961×10^{-8} W·m^2·K^{-4}

1.2 The methods are intended for measurements on large surfaces when rapid measurements must be made and where a nondestructive test is desired. They are particularly useful for production control tests.

2. Summary of Methods

2.1 At least two different types of instruments are commercially available for performing this measurement. One type measures radiant energy reflected from the specimen (Method A),[2] and the other type measures radiant energy emitted from the specimen (Method B).[3] A brief description of the principles of operation of each method follows.

2.1.1 *Method A*—The theory employed in Method A has been described in detail by Nelson et al[4] and therefore is only briefly reviewed herein. The surface to be measured is placed against an opening (or aperture) on the portable sensing component. Inside the sensing component are two semi-cylindrical cavities which are maintained at different temperatures, one at near ambient and the other at a slightly elevated temperature. A suitable drive mechanism is employed to rotate the cavities alternately across the aperture. As the cavities rotate past the specimen aperture, the specimen is alternately irradiated with infrared radiation from the two cavities. The cavity radiation reflected from the specimen is detected with a vacuum thermocouple. The vacuum thermocouple views the specimen at near normal incidence through an optical system which transmits radiation through slits in the ends of the cavities. The thermocouple receives both radiation emitted from the specimen and other surfaces, and cavity radiation which is reflected from the specimen. Only the reflected energy varies with this alternate irradiation by the two rotating cavities, and the detection-amplifying system is made to respond only to the alternating signal. This is accomplished by ro-

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[1] This method is under the jurisdiction of ASTM Committee E-21 on Space Simulation.
Effective May 19, 1971.

[2] A satisfactory instrument for this type of measurement is the Infrared Reflectometer Model DB 100, manufactured by Gier-Dunkle Instruments, Inc., Santa Monica, Calif.

[3] A satisfactory instrument for this type of measurement is the Model 25A Emissometer, manufactured by the Lion Research Corp., Cambridge, Mass.

[4] Nelson, K. E., Leudke, E. E., and Bevans, J. T., *Journal of Spacecraft and Rockets*, JSCRA, Vol 3, No. 5, 1966, p. 758.

tating the cavities at the frequency to which the amplifier is tuned. Rectifying contacts coupled to this rotation convert the amplifier output to a d-c signal, and this signal is read with a millivoltmeter. The meter reading must be suitably calibrated with known reflectance standards to obtain reflectance values on the test surface. The resulting data can be converted to total normal emittance by subtracting the measured reflectance from unity.

2.1.2 *Method B*—The theory of operation of Method B has been described in detail by Gaumer et al[5] and is briefly reviewed as follows: The surface to be measured is placed against the aperture on the portable sensing component. Radiant energy which is emitted and reflected from the specimen passes through a suitable transmitting vacuum window and illuminates a thermopile. The amount of energy reflected from the specimen is minimized by cooling the thermopile and the cavity walls which the specimen views. The output of the thermopile is amplified and sensed by a suitable meter. The meter reading must be calibrated with standards of known emittance.

3. Limitations

3.1 Both methods are limited in accuracy by the degree to which the emittance properties of calibrating standards are known and by the angular emittance characteristics of the surfaces being measured.

3.2 Method A is normally subject to a small error caused by the difference in wavelength distributions between the radiant energy emitted by the two cavities at different temperatures, and that emitted by a blackbody at the specimen temperature. Method B also has nongray errors since the detector is not at absolute zero temperature. The magnitude of this type of error is discussed by Nelson et al.[4]

3.3 Method A is subject to small errors which may be introduced if the orientation of the sensing component is changed between calibration and specimen measurements. This type of error results from minor changes in alignment of the optical system.

3.4 Method A is subject to error when curved specular surfaces of less than about 300-mm radius are measured. These errors can be minimized by using calibrating standards that have the same radius of curvature as the test surface.

3.5 Method A can measure reflectance on specimens which are either opaque or semi-transparent in the wavelength region of interest (about 4 to 50 μm). However, if emittance is to be derived from the reflectance data on a semi-transparent specimen, a correction must be made for transmittance losses.

3.6 Method B is subject to several possible significant errors. These may be due to (*1*) variation of the test surface temperature during measurements, (*2*) differences in temperature between the calibrating standards and the test surfaces, (*3*) changes in orientation of the sensing component between calibration and measurement, (*4*) errors due to irradiation of the specimen with thermal radiation by the sensing component, and (*5*) errors due to specimen curvature. Variations in test surface temperature severely limit accuracy when specimens that are thin or have low thermal conductivity are being measured. Great care must be taken to maintain the same temperature on the test surface and calibrating standards. Meter readings are directly proportional to the radiant flux emitted by the test surface, which in turn is proportional to the fourth power of temperature. Changes in orientation of the sensing component between calibration and test measurement introduces errors due to temperature changes of the thermopile. The relatively poor vacuum around the thermopile results in variations in convection heat transfer coefficients which are affected by orientation.

3.7 Method B is limited to emittance measurements on specimens that are opaque to infrared radiation in the wavelength region of interest (about 4 to 50 μm).

3.8 The emittance measured by Method B is an intermediate value between total-normal and total-hemispherical emittance because of the relationship between the thermocouple sensing elements and the test surface. The close proximity of the thermopile to the relatively large test surface allows it to receive radiation emitted over a significant angle (up

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[5] Gaumer, R. E., Hohnstreiter, G. F., and Vanderschmidt, G. F., "Measurement of Thermal Radiation Properties of Solids," *NASA SP-31*, 1963, p. 117.

to 80 deg). This error (the difference between total-normal and total-hemispherical emittance can be as large as 10 percent on certain types of specimens (such as specular metal surfaces).

4. Procedure

4.1 Calibration procedures for both methods of measurement are jointly discussed because of their similarity. In Method A infrared reflectance properties of calibrating standards must be known, and for Method B emittance values of standards are utilized. Following an appropriate warm-up time, calibrate the readout meter. Adjust the meter to give the correct reading when measuring both high and low emittance (or reflectance) standards. Repeat calibration of the meter several times at short time intervals until the correct readings can be obtained near each end of the scale. Typical high and low emittance (low and high reflectance) standards may consist of black paint (or preferably a blackbody cavity) and polished high-purity aluminum, respectively. Measure the thermal radiation properties of the standards independently with an absolute instrument, and maintain the standards in a clean condition thereafter.

4.2 In Method B care must be taken to prevent stray radiant energy from entering the sensor. This can occur if the test surface is not sufficiently flat or is not opaque.

4.3 In Method B the test surfaces and calibrating standards must be maintained at the same temperature. If thin (less than about 0.7 mm thick) conducting specimens are to be measured, they should be bonded to a thick metallic substrate. Specimen temperature changes can be noted by observing whether the indicated meter reading drifts with time.

4.4 In Method B the orientation of the sensor must be the same for both calibration and test surface measurements.

4.5 After the meter has been properly calibrated, place the test surface over the aperture of the measuring instrument. The resulting meter reading of Method A is then the infrared reflectance for blackbody radiant energy at near room temperature, or in Method B, a meter reading that can be converted to emittance using the manufacturers emittance/meter reading conversion data. In Method A, obtain the emittance by subtracting the reflectance from unity. It is recommended that the instrument be recalibrated as soon as possible after measuring the test surface. If the meter calibration has changed, repeat the entire calibration and readout procedure. It is recommended that at least three readings be taken for each test specimen, and the results averaged, to minimize statistical errors. It is also recommended that both laboratory and working emittance (or reflectance) standards be maintained, and that they be kept clean.

5. Report

5.1 The report shall include the following:

5.1.1 Name and pertinent other identification of the test material,

5.1.2 Name and pertinent other identification or traceability of the surfaces used for calibration,

5.1.3 Emittance (or reflectance) values assumed for calibration surfaces,

5.1.4 Locations on the surface area at which emittance (or reflectance) measurements were performed. (Not applicable for small individual test specimens.),

5.1.5 Ambient temperature,

5.1.6 For Method A the indicated meter reading (reflectance) shall be recorded for three successive measurements. An average of the three values shall than be calculated and subtracted from one to obtain the emittance,

5.1.7 For Method B the indicated meter reading shall be recorded for three successive measurements. These meter readings shall be converted to emittance using the manufacturer's data, and then averaged, and

5.1.8 Date and time the measurements were taken.

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Document Name: ASTM E424: Test for Solar Energy Transmittance and Reflectance (Terrestrial) of Sheet Materials

CFR Section(s): 24 CFR 200, Subpart S

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
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Designation: E 424 – 71

Standard Methods of Test for
SOLAR ENERGY TRANSMITTANCE AND REFLECTANCE (TERRESTRIAL) OF SHEET MATERIALS[1]

This Standard is issued under the fixed designation E 424; 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.

1. Scope

1.1 These methods cover the measurement of solar energy transmittance and reflectance (terrestrial) of materials in sheet form. Method A, using a spectrophotometer, is applicable for both transmittance and reflectance and is the referee method. Method B is applicable only for measurement of transmittance using a pyranometer in an enclosure and the sun as the energy source. Specimens for Method A are limited in size by the geometry of the spectrophotometer while Method B requires a specimen 0.61 m (2 ft) square. For the materials studied by the drafting task group, both methods give essentially equivalent results.

2. Applicable Documents

2.1 *ASTM Standards:*

E 259 Recommended Practice for Preparation of Reference White Reflectance Standards[2]

E 275 Recommended Practice for Describing and Measuring Performance of Spectrophotometers[2]

E 284 Definitions of Terms Relating to Appearance of Materials[2]

E 308 Recommended Practice for Spectrophotometry and Description of Color in CIE 1931 System[2]

3. Summary of Methods

3.1 *Method A*—Measurements of spectral transmittance, or reflectance *versus* a magnesium oxide standard, are made using an integrating sphere spectrophotometer over the spectral range from 350 to 2500 nm. The illumination and viewing mode shall be normal-diffuse or diffuse-normal. The solar energy transmitted or reflected is obtained by integrating over a standard solar energy distribution curve using weighted or selected ordinates for the appropriate solar-energy distribution. The distribution at sea level, air mass 2, is used.

3.2 *Method B*—Using the sun as the source and a pyranometer as a detector the specimen is made the cover of an enclosure with the plane of the specimen perpendicular to the incident radiation; transmittance is measured as the ratio of the energy transmitted to the incident energy. (The apparatus of Method B has been used for the measurement of solar-energy reflectance but there is insufficient experience with this technique for standardization at present.)

4. Significance

4.1 Solar-energy transmittance and reflectance are important factors in the heat admission through fenestration, most commonly through glass or plastics. (See Appendix A3.) These methods provide a means of measuring these factors under fixed conditions of incidence and viewing. While the data may be of assistance to designers in the selection and specification of glazing materials, the solar-energy transmittance and reflectance are not sufficient to define the rate of heat transfer

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[1] These methods are under the jurisdiction of ASTM Committee E-12 on Appearance of Materials.
Effective April 15, 1971.

[2] *Annual Book of ASTM Standards*, Part 30.

without information on other important factors. The methods have been found practical for both transparent and translucent materials as well as for those with transmittances reduced by highly reflective coatings. Method B is particularly suitable for the measurement of transmittance of inhomogeneous, patterned, or corrugated materials since the transmittance is averaged over a large area.

5. Definitions

5.1 *solar absorptance*—the ratio of absorbed to incident radiant solar energy (equal to unity minus the reflectance and transmittance).

5.2 *solar admittance*—solar heat transfer taking into account reradiated and convected energy.

5.3 *solar energy*—for these methods the direct radiation from the sun at sea level over the solar spectrum as defined in 4.2, its intensity being expressed in watts per unit area.

5.4 *solar reflectance*—the percent of solar radiation (watts/unit area) reflected by a material.

5.5 *solar spectrum*—for the purposes of these methods the solar spectrum at sea level extending from 350 to 2500 nm.

5.6 *solar transmittance*—the percent of solar radiation (watts/unit area) transmitted by a material.

6. Method A—Spectrophotometric Method

6.1 *Apparatus:*

6.1.1 *Spectrophotometer*—An integrating sphere spectrophotometer, by means of which the spectral characteristics of the test specimen or material may be determined throughout the solar spectrum. For some materials the spectrum region from 350 to 1800 nm may be sufficient. The design shall be such that the specimen may be placed in direct contact with the sphere aperture for both transmission and reflection, so that the incident radiation is within 6 deg of perpendicularity to the plane of the specimen.[3]

6.1.2 *Standards:*

6.1.2.1 For transmitting specimens, incident radiation shall be used as the standard relative to which the transmitted light is evaluated. Paired reflecting standards are used, prepared in duplicate as described below.

6.1.2.2 For reflecting specimens, use smoked magnesium oxide (MgO) as a standard as the closest practicable approximation of the completely reflecting, completely diffusing surface for the region from 300 to 2100 nm. The preferred standard is a layer (at least 2.0 mm in thickness) freshly prepared from collected smoke of burning magnesium (Recommend Practice E 259). Pressed barium sulfate ($BaSO_4$) or MgO are not recommended because of poor reflecting properties beyond 1000 nm.

6.1.3 *Specimen Backing for Reflectance Measurement*—Transparent and translucent specimens shall be backed by a light trap or a diffusing black material which is known to absorb the near infrared. The backing shall reflect no more than 1 percent at all wavelengths from 350 to 2500 nm as determined using the spectrophotometer.[4]

6.2 *Test Specimens:*

6.2.1 Opaque specimens shall have at least one plane surface; transparent and translucent specimens shall have two surfaces that are essentially plane and parallel.

6.2.2 Comparison of translucent materials is highly dependent on the geometry of the specific instrument being used. It is recommended that the specimen be placed in direct contact with the sphere to minimize and control loss of scattered radiation.

6.2.3 For specularly reflecting specimens the sphere conditions, especially where the reflected beam strikes the sphere wall, shall be known to be highly reflecting (95 percent or higher). It is recommended that a freshly coated sphere be used especially when measuring translucent or specularly reflecting specimens.

6.3 *Calibration:*

6.3.1 *Photometric*—The calibration of the photometric scale shall be done as recommended by the manufacturer. It shall be carefully executed at reasonable time intervals to ensure accuracy over the entire range.

6.3.2 *Wavelength*—Periodic calibrations should be made of the wavelength scales. Pro-

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[3] The Beckman DK-2 Recording Spectrophotometer available from Beckman Instruments, Inc., Fullerton, Calif., and the Cary 14 and 17 Recording Spectrophotometer available from Varian Assoc., Palo Alto, Calif., have been found satisfactory for this purpose. For additional apparatus specifications see Recommended Practice E 308.

[4] A piece of velvet sprayed with Nextel velvet coating 101-C10 Black available from 3M Company; Parson's Black available from Eppley Laboratories, Newport, R. I.; or Krylon Flat Black have been found satisfactory for this purpose.

cedures for wavelength calibration may be found in Recommended Practice E 275. A didymium filter has also been used for this purpose. Although the absorption peaks have been defined for specific resolution in the visible spectrum it also has peaks in the near infrared; however, the wavelength of the peaks must be agreed upon, using a specific instrument.

6.4 *Procedure:*

6.4.1 *Transmittance*—Obtain spectral transmittance data relative to air. For measurement of transmittance of translucent specimens, place freshly prepared matched smoked MgO surfaces at the specimen and reference ports at the rear of the sphere (Note 1). The interior of the sphere should be freshly coated with MgO and in good condition.

NOTE 1—Magnesium oxide standards may be considered matched if on interchanging them the percent reflectance is altered by no more than 1 percent at any wavelength between 350 and 1800 nm.

6.4.2 *Reflectance*—Obtain spectral directional reflectance data relative to MgO. Include the specular component in the reflectance measurement. Back the test specimen with a black diffuse surface if it is not opaque. Depending on the required accuracy, use the measured values directly or make corrections for instrumental 0 and 100 percent lines (see Recommended Practice E 308).

6.5 *Calculation*—Solar energy transmittance or reflectance is calculated by integration. The distribution of solar energy as reported by Parry Moon[5] for sea level and air mass 2 shall be used.

6.5.1 *Weighted Ordinates*—Obtain the total solar energy transmittance, T_{se}, and reflectance, R_{se}, in percent, by integrating the spectral transmittance (reflectance) over the standard solar energy distribution as follows:

$$T_{se} \text{ or } R_{se} = \sum_{\lambda = 350 \text{ nm}}^{\lambda = 2100 \text{ nm}} T_\lambda \text{ (or } R_\lambda) \times E_\lambda$$

E_λ for air mass 2, at 50-nm intervals, normalized to 100, is given in Appendix A1.

6.5.1.1 This integration is easily programmed for automatic computation.

6.5.2 *Selected Ordinates*—Integration is done by reading the transmittance or reflectance at selected wavelengths and calculating their average. Appendix A2 lists 20 selected ordinates for integration.[6]

6.6 *Report*—The report shall include the following:

6.6.1 Complete identification of the material tested, and whether translucent, clear, or specularly reflecting,

6.6.2 Solar T percent or Solar R percent, or both, to the nearest 0.1 percent,

6.6.3 Specimen thickness,

6.6.4 Identification of the instrument used, and

6.6.5 Integration method.

7. Method B—Pyranometer Method

NOTE 2—The pyranometer is used to measure total global (sun and sky) radiation (previously designated a 180 deg pyroheliometer; presently the latter word refers to a normal incidence measurement of direct solar radiation). See IGY Instruction Manual, Part VI, *Radiation Instruments*, Pergamon Press, New York, N. Y.

7.1 *Apparatus:*

7.1.1 *Enclosure*—The apparatus that has been used successfully is a box capable of supporting a 0.61-m (24-in.) square specimen. The box, which would normally be about 0.66-m (26-in.) square outside, should be capable of being faced in any direction, as on a universal mount. The inside of the box should be painted flat black.[4] A typical unit is shown in Fig. 1.

7.1.2 *Sensor:*

7.1.2.1 The sensing element of this instrument is a pyranometer consisting of concentric rings, or wedges of thermopiles, colored alternately black and white.[7] The voltage output of this sensor is proportional to the intensity of the total incident solar irradiation. The spectral sensitivity of this instrument extends from the ultraviolet to infrared wavelengths (280 to 2800 nm), thus encompassing all the solar spectrum. The pyranometer should be located inside the box so that the sensing thermopile is approximately 50 mm (2 in.) from the center of the bottom plane of the sample.

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[5] *Journal of the Franklin Institute*, Vol 230, 1940, p. 583, or *Smithsonian Physical Tables*, Table 1, Vol 815, 1954, p. 273.

[6] Olson, O. H., "Selected Ordinates for Solar Absorptivity Calculations," *Applied Optics*, Vol 2, No. 1, January 1963.

[7] An Eppley 50-Junction Pyranometer, Serial No. 9624 (6.66 MV/cal·cm²·min) and an Eppley 10-Junction Pyranometer, Serial No. 8553 (2.21 MV/cal·cm²·min) available from Eppley Laboratories, Inc., Newport, R. I., have been found satisfactory for this purpose. Various other Eppley Pyranometers have also been successfully used.

7.1.2.2 The pyranometer has a viewing area of 180 deg. An Eppley pyranometer with its 25-mm (1-in.) diameter sensing disk, when placed in the center of the box, views the midpoint of the edges of the test specimen as a cone of 160 deg; the diagonal of the specimen is viewed as a cone of 166 deg when the thermopile is 50 mm (2 in.) below the bottom of the specimen.

7.2.3 *Read-Out Instrumentation*—A recorder, or a nonrecording meter capable of indicating in the 0.2 to 15-mV range are permissible for use. The output voltage of the pyranometer will be affected by the input impedance of the meter to which it is connected. Thus, the meter used to indicate solar intensity should have a very high input impedance, such as a precision vacuum-tube voltmeter, or a meter which has been calibrated for one particular sensing element, thus compensating for any loading effects on that element.

7.2 *Specimens*—The test specimens should be not less than 0.61 by 0.61 m (24 by 24 in.). If the cross-sectional shape of the specimen is not flat, care must be taken to prevent the possibility of light leaks at the edges such as are caused by the use of oversize specimens.

7.3 *Procedure:*

7.3.1 Conduct the tests on a clear sunny day with no cloud cover interruptions during the individual tests. Conduct testing between the hours of 9 a.m. and 3 p.m. local standard time; this is when the solar radiation is at least 80 percent of the value obtained at solar noon for that day. In the Northern hemisphere take readings between November and February only between 10 a.m. and 2 p.m. Expose the test specimen approximately normal to the sun for 15 min prior to testing. Next, align the box normal to the sun's rays and take the average incident solar-energy reading over a period of time (normally several minutes) until a steady trace, or reading is obtained. Then place the test specimen on the box and again record the average solar energy reaching the sensor. When the test specimen has a corrugated or irregular surface move it across the sensing element, and take readings at 10-mm (½-in.) intervals for the width of one corrugation or irregularity, and average the readings. Also measure corrugated specimens with the corrugations in the North - South direction and in the East - West direction.

7.3.2 The solar energy transmittance of the test specimens is the ratio of the energy measured when the test specimen is placed between the sun and the sensor and the energy measured by the sensor with no test specimen in place.

7.4 *Report*—The report shall include the following:

7.4.1 The source and identity of the test specimen,

7.4.2 A complete description of the test specimen, that is, thickness, cross-sectional shape, color, size, translucent or transparent, type of material,

7.4.3 The percent solar energy transmittance to the nearest 1 percent,

7.4.4 The place, date, and time of the test,

7.4.5 The intensity of the solar radiation,

7.4.6 Type of sensing unit used, and

7.4.7 Ambient air temperature.

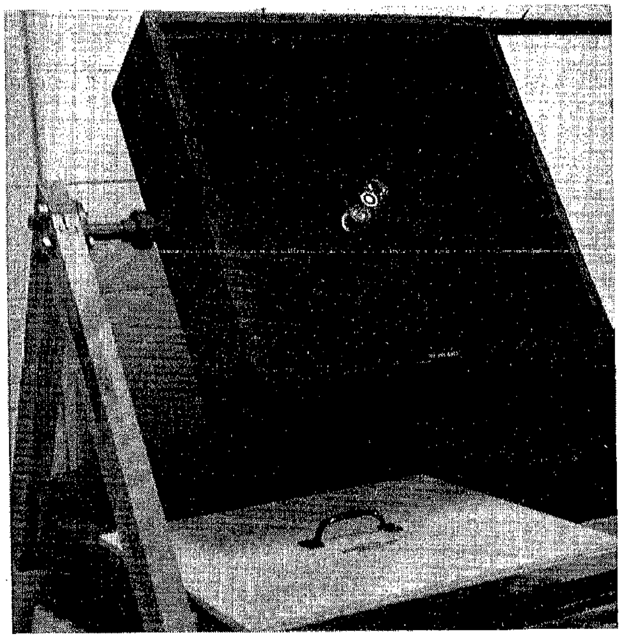

FIG. 1 Typical Unit with Pyranometer Mounted in Black Box.

APPENDIXES

A1. SOLAR ENERGY TRANSMITTANCE OR REFLECTANCE USING WEIGHTED ORDINATES (NORMALIZED TO Σ = 100.00)

$$T_{se}\ (\text{percent}) = \sum_{\lambda = 350\ \text{nm}}^{\lambda = 2100\ \text{nm}} T_\lambda \times E_\lambda$$

Wavelength, nm	Relative Energy	Wavelength, nm	Relative Energy	Wavelength, nm	Relative Energy
350	1.27	950	3.29	1550	1.49
400	3.18	1000	4.25	1600	1.36
450	6.79	1050	3.72	1650	1.17
500	8.20	1100	1.70	1700	0.89
550	8.03	1150	1.46	1750	0.54
600	7.88	1200	2.52	1800	0.01
650	7.92	1250	2.21	1850	0.00
700	7.48	1300	1.78	1900	0.00
750	5.85	1350	0.12	1950	0.12
800	5.79	1400	0.00	2000	0.02
850	5.66	1450	0.16	2050	0.26
900	3.24	1500	1.06	2100	0.58
				Total	100.00

A2. TWENTY SELECTED ORDINATES FOR EVALUATION OF SOLAR TRANSMITTANCE OR REFLECTANCE AT SEA LEVEL

No.	Wavelength, nm	No.	Wavelength, nm
1	390	11	745
2	444	12	786
3	481	13	831
4	511	14	877
5	543	15	959
6	574	16	1026
7	606	17	1105
8	639	18	1228
9	669	19	1497
10	705	20	1722

A3. SOLAR ADMITTANCE PARAMETERS

A3.1 Solar energy poses a complex problem to architects and engineers concerned with maintaining a comfortable indoor space condition. The problem exists when solar energy is admitted into a space which must be thermally and optically controlled, that is, temperature, humidity, and brightness.

A3.2 The amount of solar-energy admitted into a space can be calculated with the solar-admittance parameters, total solar-energy transmittance (TSET), and total solar-energy reflectance (TSER) of the materials surrounding the space.

A3.3 With homogenous materials the percent of solar energy reflected, R, absorbed, A, and transmitted, T, can be determined by the following equation:

$$100 \text{ percent} = R + A + T$$

A3.4 For transparent materials, such as glass and clear plastics, the total solar energy transmittance is significant and environmental control systems must be designed to handle the changing solar load.

A3.5 Space environmental engineers use the total solar-energy transmittance and total solar-energy reflectance parameters of materials to determine the solar energy admitted into a space.

A3.5.1 *For example:*

A ¼-in. bronze-tinted glass has the following typical solar energy admittance properties:

$$\text{TSET} = 46 \text{ percent} = T$$
$$\text{TSER} = 6 \text{ percent} = R$$
$$\text{TSEA} = 48 \text{ percent} = A$$

For the following conditions:

Design day—Sept. 21, 40 deg North latitude, 4 p.m., West elevation (*ASHRAE Handbook of Fundamentals*, 1967, Table 4, p. 472).[8] (All solar energy rates are per hour, square foot of glazing area.)

Direct normal solar irradiation:	230 Btu
Recommended outdoor wind velocity: (Table 9, Item 3, p. 477)[8]	7.5 mph
Corresponding outdoor surface coefficient:	4.0 Btu/deg F
Recommended indoor air velocity: (Table 9, Item 3, p. 477)[8]	Still
Corresponding indoor surface coefficient:	1.46 Btu/ deg F

Total solar energy admitted indoors:

Total solar heat gain indoors:

$$= 0.46\ (230) + 1.46/[(1.46 + 4)\ (0.48 \times 230)]$$

where:

$$0.46\ (230) = \text{transmitted solar energy} = 106 \text{ Btu}$$

and:

$$1.46/[(1.46 + 4.0)\ (0.48 \times 230)]$$

(*ASHRAE Handbook*, 1967, p. 480) Eq 19

= portion of absorbed solar energy reradiated and convected indoors

= 30 Btu (Note A1)

Total solar energy admitted indoors

= 136 Btu (Note A2)

A3.5.2 The 1967 *ASHRAE Handbook of Fundamentals*[8] reviews this procedure on pages 477 through 480.

NOTE A1—The amount of absorbed solar energy which is reradiated and convected indoors is a direct function of the air movement over the indoor and outdoor glazing surfaces.

NOTE A2—Cooling loads used for design also include conduction resulting from out-in temperature differences; heat capacity of building materials may introduce a delay in peak load timing.

A3.6 The Handbook[8] also illustrates a more commonly used method on pages 470 through 476 with the use of shading coefficients for the glazing under consideration and solar heat gain factors (SHGF) for ⅛-in. clear glass (Tables 2 to 6, pp. 470–474).

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[8] *ASHRAE Handbook of Fundamentals*, American Society of Heating, Refrigerating, and Air Conditioning Engineers, 345 E. 47th St., New York, N. Y. 10017, 1967, pp. 470–480.

A3.6.1 *For example:*

A ¼-in. bronze-tinted glass typical shading coefficient = 0.67.

If, SHGF = 205 Btu (Table 4, Sept. 21, p. 472, West elevation, 4 p.m.),

Solar energy admitted indoors = 0.67 × 205 = 137 Btu.

A3.7 Double glazing computations become more complex and the solar energy admitted indoors requires considerably more calculation as the *R A T* formula does not apply directly. For this type of glazing, the shading coefficient technique is more applicable.

A3.8 Representative shading coefficients are available from glass and plastic manufacturers.

By publication of this standard no position is taken with respect to the validity of any patent rights in connection therewith, and the American Society for Testing and Materials does not undertake to insure anyone utilizing the standard against liability for infringement of any Letters Patent nor assume any such liability.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

Document Name:	ASTM E606: Standard Recommended Practice for Constant-Amplitude Low-Cycle Fatigue Testing
CFR Section(s):	24 CFR 200.946
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OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 606 – 80

An American National Standard

Standard Recommended Practice for
CONSTANT-AMPLITUDE LOW-CYCLE FATIGUE TESTING[1]

This standard is issued under the fixed designation E 606; 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 recommended practice covers the determination of low-cycle fatigue properties of nominally homogeneous metallic materials by the use of uniaxially loaded test specimens. It is intended as a guide for low-cycle fatigue testing performed in support of such activities as materials research and development, mechanical design, process and quality control, product performance, and failure analysis.

1.2 The use of this recommended practice is limited to metallic specimens and does not cover testing of full-scale components, structures, or consumer products.

1.3 This recommended practice is applicable to temperatures and strain rates for which the magnitudes of time-dependent inelastic strains are negligible relative to the magnitudes of time-independent inelastic strains. No restrictions are placed on environmental factors, such as temperature, pressure, humidity, medium, etc., provided they remain constant throughout the test and do not cause loss of or change in dimensions with time.

NOTE 1—The term *inelastic* is used herein to refer to all nonelastic strains. The term *plastic* is used herein to refer only to the time-independent (that is, noncreep) component of inelastic strain.

NOTE 2—Appendix X4 has been added to cover those test conditions where time dependent strains may be present.

1.4 This recommended practice is restricted to the testing of axially loaded uniform and hour-glass profile test specimens in the low-cycle fatigue regime. Testing is limited to constant-amplitude stress or strain cycling in which stress and strain are to be continuously varying (that is, without hold times).

2. Applicable Documents

2.1 *ASTM Standards:*

A 370 Methods and Definitions for Mechanical Testing of Steel Products[2]

E 3 Methods of Preparation of Metallographic Specimens[3]

E 4 Practices for Load Verification of Testing Machines[4]

E 8 Methods of Tension Testing of Metallic Materials[5]

E 9 Methods of Compression Testing of Metallic Materials at Room Temperature[6]

E 83 Practice for Verification and Classification of Extensometers[6]

E 111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus[6]

E 112 Methods for Determining Average Grain Size[3]

E 132 Test Method for Poisson's Ratio at Room Temperature[6]

E 157 Method of Assigning Crystallographic Phase Designations in Metallic Systems[3]

E 209 Practice for Compression Tests of Metallic Materials at Elevated Temperatures with Conventional or Rapid Heating Rates and Strain Rates[6]

E 337 Test Method for Measuring Humidity with a Psychrometer (The Measurement of Wet-Bulb and Dry-Bulb Temperatures)[7]

E 384 Test Method for Microhardness of Materials[8]

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[1] This recommended practice is under the jurisdiction of ASTM Committee E-9 on Fatigue, and is the direct responsibility of Subcommittee E09.08 on Fatigue Under Cyclic Strain.

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[2] *Annual Book of ASTM Standards*, Vols 01.01, 01.02, 01.03, 01.04, 01.05, and 03.01.

[3] *Annual Book of ASTM Standards*, Vol 03.03.

[4] *Annual Book of ASTM Standards*, Vols 03.01, 04.02, 07.01, and 08.03.

[5] *Annual Book of ASTM Standards*, Vols 02.01, 02.02, 02.03, and 03.01.

[6] *Annual Book of ASTM Standards*, Vol 03.01.

[7] *Annual Book of ASTM Standards*, Vols 07.01, 11.03, and 15.09.

[8] *Annual Book of ASTM Standards*, Vols 03.01, 03.03, and 03.04.

E 399 Test Method for Plane-Strain Fracture Toughness of Metallic Materials[6]

E 467 Practice for Verification of Constant Amplitude Dynamic Loads in an Axial Load Fatigue Testing Machine[6]

E 513 Definitions of Terms Relating to Constant-Amplitude Low-Cycle Fatigue Testing[6]

3. Significance

3.1 Although low-cycle fatigue is a phenomenon that is influenced by many of the same variables that influence high-cycle fatigue, the nature of low-cycle fatigue imposes distinctive requirements on fatigue testing methods. In particular, cyclic total strain should be measured and cyclic plastic strain should be determined. Furthermore, either of these strains typically are used to establish cyclic limits; total strain usually is controlled throughout the cycle. The uniqueness of this recommended practice and the results it yields is the determination of cyclic stresses and strains throughout the tests.

3.2 Low-cycle fatigue can be an important consideration in the design of industrial products. It is important for situations in which components or portions of components undergo either mechanically or thermally induced cyclic plastic strains that cause failure within relatively few cycles. Information obtained from low-cycle fatigue testing may be an important element in the establishment of design criteria to protect against component failure by fatigue.

3.3 Low-cycle fatigue test results are useful in the areas of mechanical design as well as materials research and development, process and quality control, product performance, and failure analysis. Results of a low-cycle fatigue test program may be used in the formulation of empirical relationships between the cyclic variables of stress, total strain, plastic strain, and fatigue life. They are commonly used in data correlations such as curves of cyclic stress or strain versus life and cyclic stress versus cyclic plastic strain. Examination of the cyclic stress - strain curve and its comparison with monotonic stress - strain curves gives useful information regarding the cyclic stability of a material, for example, whether the values of hardness, yield point, yield strength, and strain hardening exponent will increase or decrease (that is, whether a material will harden or soften) due to cyclic plastic straining. Results of the uniaxial tests on specimens of simple geometry can be applied to the design of components with notches or other complex shapes provided that the strains can be determined and multiaxial states of stress or strain and their gradients are correctly correlated with the uniaxial strain data.

4. Definitions

4.1 The definitions in this recommended practice are in accordance with Definitions E 513.

5. Functional Relationships

5.1 Empirical relationships that have been used commonly for description of low-cycle fatigue data are given in Appendix X1.

5.2 Relationships for conversion of diametral strain to axial strain are given in Appendix X2.

6. Apparatus

6.1 *Testing Machine*—Testing should be conducted with a tension - compression fatigue testing machine that conforms to the requirements of Practices E 4 and Practice E 467. The machine should be one in which specific measures have been taken in its design to minimize backlash in the loading train.

NOTE 3—Load capacity of 45 kN (approximately 10 kips) or greater would be sufficient for the recommended specimens (Section 7) and most test materials. The machine load capacity used for these specimens ordinarily would not exceed 110 kN (approximately 25 kips).

6.2 *Stress or Strain Control*—Testing machine controls should permit cycling between constant stress or strain limits. If material behavior permits (for example, aging effects do not hinder), control stability should be such that the respective stress or strain limit is repeatable from cycle-to-cycle to within 0.5 % of the average control limit and repeatable over the test duration to within 2 % of the average control limit.

6.3 *Fixtures*—Fixtures should be provided for rigidly gripping specimen ends and accurately transmitting cyclic loads (without backlash) along the longitudinal specimen axis.

6.3.1 To minimize bending strains, specimen fixtures should be aligned such that the major axis of the specimen closely coincides with the load axis throughout each loading cycle. It is important that the accuracy of alignment be kept consistent from specimen to specimen. Alignment should be checked by means of a trial test specimen with longitudinal strain gages placed at three or four equidistant locations around the minimum diameter. The trial test specimen should be turned about its axis, installed, and checked for each of three or four orientations within the fixtures. The maximum bending strains so determined should not exceed 5 % of the minimum axial strain range imposed during any test program. For specimens having a uniform gage length, it is advisable to place a similar set of gages at two or three axial positions within the gage section. One set of strain gages should be placed at the center of the gage length to detect misalignment that causes relative rotation of the specimen ends about axes perpendicular to the specimen axis. An additional set of gages should be placed away from the gage-length center to detect relative lateral displacement of the specimen ends.

6.3.2 Several commonly used fixturing techniques are shown schematically in Fig. 4. The selection of any one fixturing technique depends primarily upon the user's specimen design. The fixtures should be constructed of hardened steel for high strength and abrasion resistance. Chrome or nickel plating may be needed for corrosion resistance. Fixtures may be coupled with the Wood's metal pot **(1, 2)**[10] of Fig. 3 to permit initially zero specimen stress during fixturing. Placement of the fixtures within a die-set or flex bars alleviates relative lateral motion of specimen ends and provides rigidity that is important in preventing compressive buckling of the test specimen. Initially, zero fixturing stress combined with precise and rigid alignment may be achieved by installing fixtures and a Wood's metal pot within the die-set or flex bars.

6.4 *Extensometers*—Extensometers should be employed for the purpose of measuring deformation in the gage section. They should be suitable for dynamic measurements over long periods of time.

6.4.1 The extensometers may be of two major types: electromechanical (for example, the more frequently used strain gage or LVDT type as shown in Fig. 5) or optical (for example, using image orthicon tubes **(2)** or lasers). In all cases, they should generate information suitable for readout and recordings. The extensometers should qualify as Class B-2 or better in accordance with Practice E 83; however, error in measured value of strain should not exceed ± 2.5 % of strain amplitude.

6.4.2 Extensometers should measure longitudinal deformation when a uniform-gage specimen, such as shown in Fig. 1(*a*), is tested. Generally, these extensometers are attached as shown in Fig. 5(*a*). Either the strain-gage or LVDT systems of Figs. 5(*c*) and 5(*d*) may be used with the probes shown in Fig. 5(*a*).

NOTE 4—Care should be exercised when installing the longitudinal extensometer so as to prevent damage to the specimen surface and consequential premature fatigue failure at the gage points. Small strips of cellophane tape can be applied to the specimen surface where the extensometer knife edges make contact so that the knife edges imbed in the tape rather than in the specimen. However, use care to ensure that no creep or slippage occurs in the tape adhesive.

6.4.3 Extensometers should measure diametral deformations when specimens having hourglass profiles are tested. A typical method of diametral displacement measurement is shown in Fig. 5(*b*). Curved extensometer tips, convex in the longitudinal plane, can provide point contact during testing. Either of the strain-gage or LVDT systems of Figs. 5(*c*) or 5(*d*) may be used with the probes shown in Fig. 5(*b*). Care should be exercised during installation of the diametral extensometer to prevent damage to the test specimen surface. Extensometer tips should be adjusted properly to minimize the force they impose on the specimen. When installing the extensometer, gently move its tip longitudinally along the specimen while watching the gage readout to find the minimum diameter. Calibration of extensometers should be conducted before and after each test program.

NOTE 5—Care should be taken in the measurement of diametral strains for materials such as cast materials that possess large crystals and a large degree of preferred orientation. These, as well as hexagonal close-packed materials, tend to be anisotropic and may require special methods of strain measurement and interpretation.

6.5 *Load Transducers*—A load transducer should be placed in series with the test specimen for the purpose of measuring magnitude and

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[10] The boldface numbers in parentheses refer to the references at the end of this standard.

sense of the axial load transmitted through the specimen. It should be designed specifically for fatigue testing and possess the following characteristics: high resistance to bending; low axial compliance; high linearity, accuracy, and sensitivity; low hysteresis. Load transducer capacity should be selected to adequately cover the range of loads to be observed, but not so large as to render large errors (that is, greater than 1 % peak measured load) in the measurement of small loads. Load transducer calibration should be verified in accordance with Practices E 4 and E 467.

6.6 *Recording Systems*—The following recording systems should be considered a minimum requirement. Other data-retrieval systems, such as those incorporating digital computers, provide an acceptable substitute for the analog systems named here. Accuracy of recording systems should be kept within 1 % of full scale or close thereto in the case of oscilloscopes or oscillographic recorders.

6.6.1 *X-Y Recording*—Some means of *X-Y* recording should be used for the purpose of recording hysteresis loops of load versus deformation or stress versus strain. A potentiometric *X-Y* recorder or an oscilloscope equipped with a camera are acceptable alternatives. The potentiometric *X-Y* recorder should be used only when the rate of cycling results in a pen velocity that is less than one half of the recorder's slewing speed. At higher frequencies, the oscilloscope should be used.

6.6.2 *Strip Chart Recording*—Strip chart recorders should be available to monitor load (or strain). The frequency of strain cycling should be such that recording pen velocity never exceeds one half of the recorder's slewing speed. It is recommended that these recorders be calibrated at the testing frequency used.

6.7 *Cycle Counter*—A cycle counter shall be available for the purpose of counting and indicating total accumulated cycles of straining. A timer is a desirable adjunct to the cycle counter. When used to indicate total elapsed time to failure, it provides an excellent check against the cycle counter and frequency.

6.8 *Strain Computer*—An analog (or digital) computer is recommended for use in low-cycle fatigue tests of hourglass specimens whenever appreciable cyclic hardening and softening occur during the test. Such a computer is useful when used in the real time mode with servo-controlled testing machines and can be used for limit control of screw-driven machines. The computer should be designed to convert diametral strain and axial load signals into an axial strain signal. See Appendix X1 for conversion relations. In the case of servo-controlled machines, this axial strain signal may be used as a feedback signal for control purposes, thus simulating axial strain control. A block diagram for the analog (or digital) computer program is shown in Fig. 6.

6.9 *Calibration*—The calibration interval of all electronic recording and transducer systems should be performed in accordance with the manufacturer's recommendations; in the absence of these, the interval should be no greater than six months and even more frequent if necessary to maintain required accuracy. Calibration should be checked whenever accuracy is in doubt.

7. Specimens

7.1 *Specimen Design*—Figure 1 shows two basic specimen configurations. Figure 1(*a*) shows a recommended uniform-gage specimen and Fig. 1(*b*) shows the recommended hourglass profile. Selection of either 1(*a*) or 1(*b*) is subject to anisotropy and buckling considerations (see Note 5). Both of these recommended specimens possess a solid circular cross section and a minimum diameter of 6.35 mm (0.25 in.) in the test section. Specific cross-sectional dimensions are listed here only because they have been predominant in the generation of the low-cycle fatigue data base that exists in the open literature. Specimens possessing other diameters or tubular cross sections may be tested successfully within the scope of this recommended practice; however, crack growth rate, specimen grain size, and other considerations might preclude direct comparison with test results from the recommended specimens. While design of specimen end connections is primarily dependent upon user preference (see Note 7), a number of commonly used configurations are shown in Figs. 1(*c*), 1(*d*), and 1(*e*). Care must be exercised in the machining of uniform-gage specimens to blend the shoulder radius at the specimen ends with minimum diameter so as to avoid undercutting. So that stress concentrations are minimized, shoulder radius should be as large as possible, consistent with limitations on specimen length.

NOTE 6—Selection of either the uniform-gage section or hourglass profile is commonly based upon the magnitude of compressive strain to be imposed. The recommended uniform gage specimen is frequently suitable for strain ranges up to about 2 %. Above 2 %, hourglass specimens are recommended. Soft materials or elevated temperatures may dictate lower strain ranges. The recommended hourglass specimen possesses a profile ratio of 12:1 for radius of curvature to minimum radius of specimen. If the user wishes, different ratios between the limits of 8:1 and 16:1 may be employed. Lower limits will increase stress concentration and may affect fatigue life; higher ratios limit the specimen's buckling resistance. For some materials tested in the low life range, hourglass specimens might give different results from similarly stressed uniform-gage specimens. When test material is anisotropic, uniform specimens are recommended. It is very difficult to determine axial strains from measurements of diametral strain in hourglass specimens for many anisotropic materials.

NOTE 7—Design of specimen end connections is dependent upon user preference, fixturing, or availability of material or a combination of all three; it is constrained principally by proper consideration of axial alignment and backlash. Button-head end connections, such as those shown in Figs. 1(*d*) and 1(*e*), permit precise alignment with a specimen end clamping preload (to avoid backlash in the grip). The threaded connection, shown in Fig. 1(*c*), is useful where the available material is not thick enough to provide for the larger diameter button-head ends. The efficiency button-head connection, shown in Fig. 1(*e*), provides the button-head preloading feature, without requiring larger diameter ends. The button-head design is useful at elevated temperatures, as it does not suffer the "oxidation-sticking" experienced with threaded ends, but it may produce some specimen failures in the fixture when used at room temperature.

7.1.1 *Alternative Specimen Design for Sheet Specimens*—Often, it is desirable to obtain test specimens from sheet material that is less than 6.35 mm (0.25 in.) thick. In general, the considerations discussed in other sections apply equally to sheet testing. However, special specimen geometries and gripping arrangements, as well as more sensitive load and strain transducers, are necessary. Typical specimen designs that have been used successfully are shown in Fig. 2. The specimen in Fig. 2(*a*) has a rectangular cross section and is suitable up to strain amplitudes of at least 1 % applied to sheets as thin as 2.54 mm (0.100 in.). For higher strain amplitudes, the cylindrical cross section hourglass specimen in Fig. 2(*b*) is recommended. See Ref. (3) for other designs.

7.2 *Specimen Preparation*—Specimens should be prepared by a specific set of procedures that is known to provide consistent test results. Agreement between the testing organization and the user of the test results concerning preparation procedures should be obtained. The following provides recommended guidelines.

7.2.1 *Specimen Coupons and Materials*—Coupons from which specimens are machined should either be nominally homogeneous or sampled from the source material, or both, so as to be statistically representative of the properties sought in the application of the material to its end use. Thus, when material requirements allow, specimens should be removed from the fabricated component of interest. Any material orientations, such as rolling direction or casting direction, should be identified with respect to the orientation of the specimen axes. Orientation notation used in accordance with Method E 399 is acceptable such that L, T, S, LT, LS, or TS refer to the longitudinal axis of the specimen.

7.2.2 *Specimen Surface Preparation*—Specimens prepared from coupons will possess a "surface preparation history" as a consequence of machining operations, heat treatments, and the effects of environment during the storage period prior to testing. Unless the purpose of testing is to determine the influence of specific surface conditions on fatigue life, it is recommended that specimen surface preparation be performed in a manner that will have a minimum influence upon the variability in fatigue lives exhibited by the specimen group tested. Ordinarily, this would be accomplished by:

7.2.2.1 Consistently machining specimens to be as smooth and uniform in surface finish (in the gage region) as feasible for the subject material and the machining techniques available, and by employing as a final operation a machining or other "finishing" procedure that would introduce minimal surface metal distortion (see Note 8), and by

7.2.2.2 Ensuring, through the use of protective atmospheres, that surface attack, such as oxidation and corrosion, does not occur, either during heat treatments or during specimen storage, for all specimens within a program.

NOTE 8—Appendix X3 presents an example of a machining procedure that has been employed on some metals to minimize variability of machining and heat treatment influences upon fatigue life.

The exact procedure of specimen preparation and handling should be carefully documented.

7.3 *Specimen Storage*—Test specimens that may be susceptible to corrosion in moist room temperature air should be protected immediately after preparation and stored until they are tested. Specimens may be stored in a suitable protective environment, such as dry inert gas (as might be conveniently employed in a laboratory desiccator) or a vacuum.

7.4 *Materials Description*—A complete material description is desirable. It is recommended that the following metallurgical and mechanical properties be obtained:

7.4.1 *Metallurgical Characteristics*—Chemical composition, grain size (see Methods E 112), crystallographic structure, preferred orientation if present, general shape of grains (that is, equiaxed or elongated), second phase particles (see Method E 157), heat treatment (whether at the mill, during fabrication, in the laboratory, or a combination of all three), and specification designation (ASTM, ASME, AISI, Military, etc.).

7.4.2 *Mechanical Properties*—For purposes of performing the test and calculating results, it is desirable to have available the following representative mechanical properties, obtained at the appropriate temperature and measured in accordance with the applicable standards such as Methods E 8, E 9, E 111, E 132, and Practice E 209: tensile or compressive yield strength or yield point, or both, ultimate tensile strength, percent elongation, percent reduction of area, Poisson's ratio, and Young's modulus. The following true stress-strain properties may also be desirable: true fracture strength, true fracture ductility, strain hardening exponent, and strength coefficient. Hardness may also be determined in accordance with Methods A 370 or Test Method E 384, or both.

8. Procedure

8.1 *Test Environment:*

8.1.1 *Temperature*—Temperatures should be low enough that the magnitudes of time-dependent inelastic strains are negligible relative to the magnitudes of time-independent inelastic strains. For the duration of testing, specimen temperature should not vary by more than ±2°C (±3.6°F). Temperature gradients should be within ±2°C over the gage length of uniform-gage specimens. Initially, monitor the specimen temperature to determine the extent of heating due to strain cycling. If necessary, provide cooling to maintain the above limits.

NOTE 9—If the temperature cannot be maintained to within ±2°C, then temperature deviations should be reported. If possible, the effect of temperature should be demonstrated throughout the range of test temperatures. Preliminary testing is desirable if the time-dependent effects are not already known.

8.1.2 Elevated temperatures may be imposed by any of several methods: (*1*) high-frequency induction, (*2*) radiant furnace, or (*3*) immersion in an inert heated gas or liquid. In (*1*) and (*2*) above, an enclosure is recommended to prevent air currents in the vicinity of the specimen from causing undesirable temperature gradients. Specimens tested at room temperature should also be in draft-free surroundings. Temperatures below room temperature may be imposed by placing the specimen and gripping apparatus in a refrigerated chamber that may be either of the liquid or gaseous type, depending on temperature requirements and other possible environmental considerations. Liquefied gases, such as liquid nitrogen, or solidified gases, such as dry ice placed in a liquid medium, provide possible means for low-temperature testing.

NOTE 10—Use of glass insulation may avoid difficulty with wires submerged in a cooling solvent.

8.1.3 If testing is performed in air, relative humidity may be measured in accordance with Method E 337, unless it has already been determined that moisture has little or no effect on fatigue life for the material under test. If an effect is present, relative humidity should be controlled; when uncontrolled, it should be carefully monitored.

8.2 *Measurement of Test Specimen Dimensions*—For the purpose of making an accurate determination of specimen cross-sectional area, measure the reduced section as follows:

8.2.1 Measure the diameter at the center of the gage section by means of an optical comparator or other optical means read or estimated to an accuracy of 0.0125 mm (0.0005 in.) or better. A precision micrometer may be used in place of the optical comparator if its use does not damage the gage section surface in a way as to affect specimen performance. For uniform-gage specimens, check diameters for at least two other positions within the specimen gage length.

8.3 *Test Machine Control*—It is necessary to control one (or more) variable(s) (for example, stress, strain, load, displacement, or other appropriate parameter) in a manner that is in keeping with the test objectives.

8.3.1 *Control Mode*—Total axial strain amplitude is the most commonly utilized control variable in a low-cycle fatigue test. Total axial strain is often controlled continuously throughout each fatigue cycle in a manner prescribed in 8.4. It is also acceptable to control only the limits of either total axial strain or plastic axial strain. In such cases vary another variable, such as diametral strain, displacement, or load, between these limits in some cyclically consistent manner under either closed loop or other control means. For long-life fatigue tests that exhibit low but measurable levels of plastic strain, it is acceptable to control load while monitoring plastic and total strain and making periodic adjustments of mean load and load range in order to maintain the desired strain limits.

8.3.2 *Closed Loop Method*—Fatigue testing machines of the closed loop servo-controlled type often are capable of continuously controlling specific test variables such as load or displacement through appropriate selection of feedback signals. Application of scale factors to these signals thereby permits continuous control of stress or strain. Axial stress may be scaled directly from the load cell signal. Axial strain may be scaled directly from an axial extensometer signal when uniform-gage specimens are tested. When hourglass specimens are tested, an axial strain signal must be determined from a diametral extensometer signal and the force signal by means of a computer (see 6.9) if closed loop control of axial strain is desired. Closed loop control of diametral strain is generally not equivalent to closed loop control of axial strain since the relationship between axial strain and induced diametral strain changes during cycling for materials that undergo cyclic hardening and softening.

8.3.3 *Other Control Methods*—Fatigue testing machines that do not provide continuous closed loop control of either specimen load or specimen displacement generally have the capability to impose limits on the chosen test variable. However, they do not control that variable throughout the fatigue cycle. Limit control is a special case of closed loop control. Thus, load and displacement signals may be handled in a manner similar to that of 8.3.2 to determine strain limits. It is not necessary to use a computer with these machines for limit control of hourglass specimens if periodic adjustments are made to the diametral strain amplitude in such a manner as to maintain constant axial strain limits. These adjustments are necessary for materials that undergo significant cyclic hardening and softening because of attendant changes in the relationship between axial strain and induced diametral strain.

8.4 *Waveform*—The strain (or stress) versus time waveform should be identical throughout a test program unless test objectives are to determine waveform effects. In the absence of specific waveform requirements or equipment limitations, a triangular waveform is preferred.

8.5 *Strain Rate and Frequency of Cycling*—Either strain rate or frequency of cycling should be held constant for the duration of each test as well as for the duration of a test program, unless the test objective is specifically to determine either strain rate or frequency effects, respectively.

NOTE 11—While constant-strain-rate testing is often preferred, constant-frequency testing may be of greater practical significance to the fatigue analysis of certain machine components. On the other hand, constant-strain-rate testing may be experimentally more tractable than constant-frequency testing since long-life, small-strain tests in the former mode may be completed in shorter periods of time than tests conducted in the latter mode.

8.5.1 If nontriangular waveforms or equipment limitations preclude constant-strain-rate testing and time limitations preclude constant-frequency testing, other means of rate control are available. One accepted procedure is to maintain constant average strain rate (twice the product of strain range and frequency) throughout each test and for the duration of the test program. Another acceptable procedure, one that is most convenient when testing under plastic strain limit control, is to maintain constant average plastic strain rate.

8.5.2 The selected range of strain rates or frequencies should be sufficiently low as to preclude specimen heating in excess of 2°C (3.6°F). In a servo-controlled testing machine, make a comparison of the program and feedback signals to ensure that the selected rates or frequencies are and remain within system capabilities and accuracy requirements. Frequency response of extensometers (depending upon their design) is often a limiting factor in the system.

NOTE 12—Notwithstanding the need for constancy of rate, the testing rate may be reduced briefly in order to permit periodic recording operations if doing so does not change specimen behavior.

8.6 Begin all tests on the same half-cycle, tensile or compressive, unless the purpose of testing is to study initial loading effects. For some materials it is acceptable to increase strain amplitude gradually and continuously over a period not greater than 2 % of anticipated life.

8.7 *Number of Specimens*—It is suggested that a minimum of ten specimens be used to generate a fatigue strain-life curve. It is also suggested that the replication guidelines given in STP 588 **(4)** be followed, especially if subsequent statistical analysis is planned.

8.8 *Recording*—Unless computerized data retrieval systems are employed continuously, record the initial series of hysteresis loops of axial stress (or load) versus total or plastic axial strain (versus total or plastic diametral strain if an axial strain signal is not available). Record single hysteresis loops thereafter at successively larger increments of cycle count. For tests of 100 cycles or more, a minimum of ten additional hysteresis loops is desirable. When practical, continuously record the dependent variables (for example, axial stress and plastic axial strain in a total axial strain control test) as a function of time.

NOTE 13—When continuous recording is not practical either due to lengthy test durations or the limited availability of recorders, intermittent records or alternative sampling of the recorded variables is acceptable.

8.9 *Determination of Failure*—Failure can be determined in a number of ways dependent on the ultimate use of fatigue life information and the nature of the material being tested. (See Note 13.) Several commonly used criteria include the following:

8.9.1 Total separation or fracture of the specimen into two parts at (*a*) some location within the gage section of a uniform-gage specimen, or (*b*) the vicinity of the minimum diameter in an hourglass specimen. Fracture at any other location than specified above should be considered as an invalid result.

NOTE 14—A post-mortem failure analysis should be performed to uncover any unusual causes of failure. Inclusions, voids, machining defects, etc., that are not representative of the bulk material or its application may render an invalid fatigue life determination. (See 8.11.3.)

8.9.2 For fully reversed straining (or loading), a drop in the peak tensile stress (or increase in tensile peak strain) that is some preselected percentage greater than a similar drop in the peak compressive stress (or increase in compressive peak strain).

8.9.3 For strain control, cusp formation in the compressive portion of the hysteresis loop, such that the size of the cusp (Note 14) has grown to some preselected percentage of the peak compressive stress.

NOTE 15—Cusp size is taken as peak compressive stress minus stress at the inflection point of the compressive loading curve (see Fig. 7). Frequent *X-Y* recordings should be made in order to observe cusp formation.

8.9.4 A change in the rate-of-change of cyclic load range (or deformation range) that exceeds some preselected percentage change.

NOTE 16—For failure criteria 8.9.2 through 8.9.4, care should be exercised in the interpretation of cyclic stress-strain behavior when hysteresis loops exhibit serrations and cyclic hardening mechanisms compete with cyclic softening mechanisms for dominance at different times throughout life.

8.9.5 The existence of surface microcracks (for example, as observed optically or by replicas) that are larger than some preselected size consistent with test objectives.

8.10 Conduct testing at least until failure and preferably until fracture when testing needs dictate and economics allow. Record total accumulated cycles to failure (and fracture) by means of a cycle counter and check against an elapsed time meter.

8.11 *Analysis of Data*—While it is not the purpose of this recommended practice to specify data analysis techniques, the following example represents a common procedure:

8.11.1 *Determination of the Cyclic Stress - Strain Curve*—Generate a cyclic stress - strain curve from paired values of stress range and plastic strain range **(5)**. When practical, assume a simplifying mathematical expression for the cyclic stress - strain relationship.

NOTE 17—For many metals, one accepted and convenient expression of the cyclic stress - strain curve is that of a power relationship presented by Eq 1 of Appendix X1.

8.11.2 *Determination of the Strain-Life Relationship*—Generate a strain-life curve from paired values of total strain versus life or plastic strain versus life. When practical, assume a simplifying mathematical relationship.

NOTE 18—For many metals, one accepted and convenient means for representing the strain-life relationship is presented by Eq 4 of Appendix X1.

8.11.3 *Post-Mortem Examinations*—Metallographic examination of the failed specimens is desirable for a variety of purposes depending in part on user interests. Of foremost importance is a fractographic examination of the two crack surfaces to determine any unusual causes of failure that might invalidate the test results. Scanning electron microscopy and transmission electron microscopy of fracture replicas are two common methods used in such an investigation. Ref. **(6)** provides a useful basis for fractographic analysis. The techniques of light metallography and transmission electron microscopy are frequently used when studying structural changes that occur during fatigue or the effects of metallurgical structure on fatigue behavior.

9. Report

9.1 The list of items of information that follows is suggested for inclusion in any report. When publishing results in the open literature, include as much information as possible, independent of the author's purpose. Routine laboratory reports need include only information pertinent to the end use of the test data. Minimum recommended requirements are indicated by an asterisk.

9.1.1 Specific objective of testing.

9.1.2 *Specimen Materials Description* (including processing):

9.1.2.1 All available mechanical properties including: yield strength or yield point, or both, ultimate tensile strength, percent elongation, percent reduction of area, Poisson's ratio, elastic modulus, true fracture strength, true fracture ductility, static strain hardening exponent, static strength coefficient, hardness number, and degree of cold work.

9.1.2.2 All available metallurgical characteristics: certified chemical composition, grain size, crystallographic structure, preferred orientation with respect to specimen axes, general shape of grains (that is, equiaxed or elongated), second phase particles, and heat treatment. Include photomicrographs when possible to document the above properties.

9.1.3 *Specimen Description:*

9.1.3.1 Drawing of specimen design*.

9.1.3.2 Specimen machining and surface preparation procedures*.

9.1.3.3 Deviations from recommended specimens and specimen preparation procedures if any.

9.1.4 *Description of Equipment:*

9.1.4.1 Specimen fixtures.

9.1.4.2 Testing machine*.

9.1.4.3 Transducer systems (that is, load transducers, deformation transducers).

9.1.4.4 Recorders and recording equipment.

9.1.4.5 Deviations from recommended equipment, if any.

9.1.5 *Description of Testing Environment*:*

9.1.5.1 Gas, liquid, or vacuum; chemical composition of medium*.

9.1.5.2 Humidity of gaseous environment*.

9.1.5.3 Test temperature and temperature control method*.

9.1.5.4 Temperature variations or deviations, or both, from those specified.

9.1.6 *Testing Conditions and Procedure:*

9.1.6.1 Deviations from recommended procedures, if any.

9.1.6.2 Frequency of cycling (or cyclic strain rate) and description of waveform*.

9.1.6.3 Mode of control, that is, load or stress, continuous strain control, strain limit control, axial strain feedback, diametral strain feedback, etc*.

9.1.6.4 Ratio of axial strain limits (minimum and maximum) and total axial strain range*.

9.1.6.5 Procedure for maintaining constant axial strain limits.

9.1.6.6 Sign of strain at first quarter cycle, tensile or compressive.

9.1.7 *Test Results*—Tabulate the results for all test specimens. When used for purposes of structural analysis, the following two items are most important:

9.1.7.1 Initial, stabilized, or half-life values, or a combination of all three, of the dependent variables from the list of stress range, strain range, and plastic strain range. Complete curves of these values throughout the specimen life. When complete curves are impractical, curves through intermittent values are acceptable*.

9.1.7.2 Total number of cycles to failure, N_f, and failure definition used in the determination of N_f*.

9.1.7.3 Order in the sequence in which each specimen was tested.

9.1.8 Results of analysis for cyclic stress - strain properties. If data analysis is performed by means of the relationships in Appendix X1, a tabulation of results should include the cyclic strain hardening exponent and the cyclic strength coefficient.

9.1.9 Results of analysis for strain-life properties. If data analysis is performed by means of the relationships in Appendix X1, a tabulation of results should include the fatigue strength exponent, the fatigue ductility exponent, the fatigue strength coefficient, and the fatigue ductility coefficient.

9.1.10 Brief description of the fracture characteristics; results of post-test metallography and scanning electron microscopy; identification of fracture mechanism and the relative degree of transgranular and intergranular cracking.

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(1) Raske, D. T., and Morrow, JoDean, "Mechanics of Materials in Low Cycle Fatigue Testing," *Manual on Low Cycle Fatigue Testing, ASTM STP 465,* Am. Soc. Testing Mats., 1969, pp. 1–25.

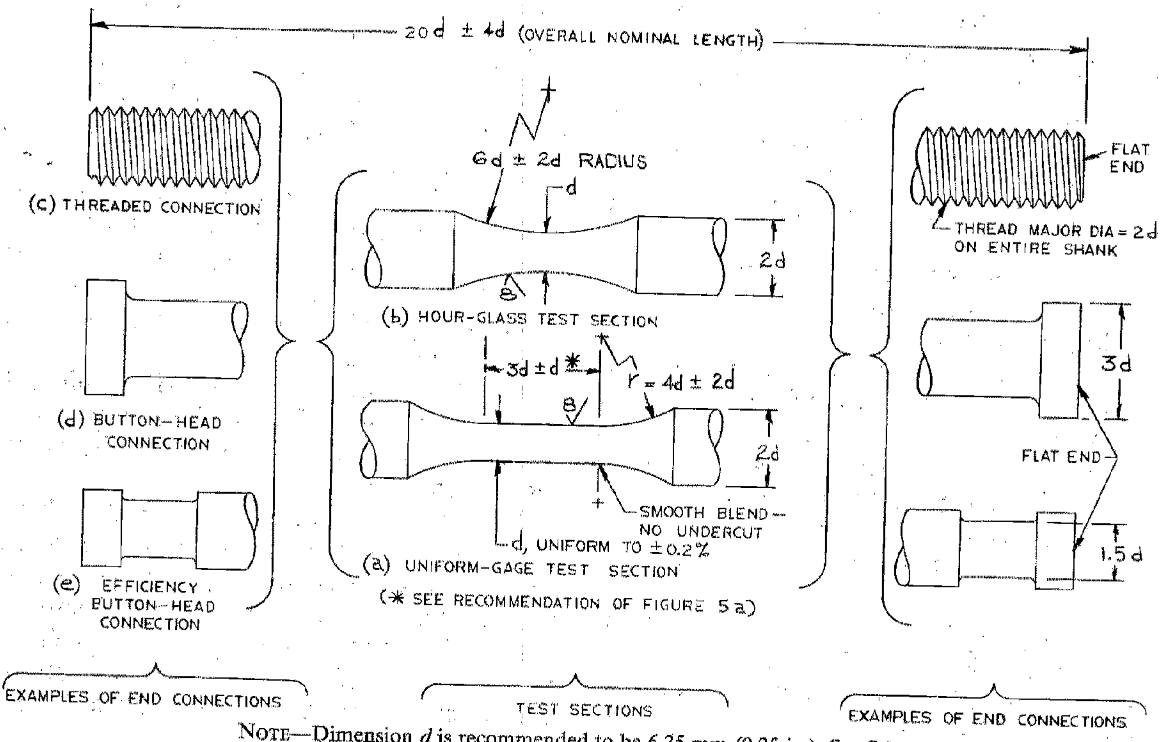
(2) Feltner, C. E., and Mitchell, M. R., "Basic Research on the Cyclic Deformation and Fracture Behavior of Materials," *idem,* pp. 27–66.

(3) Marsh, G. M., Robb, A. D., and Topper, T. H., "Techniques and Equipment for Axial Fatigue Testing of Sheet Steel," SAE Paper No. 730578, May 1973.

(4) Little, R. E., and Jebe, E. H., *Manual on Statistical Planning and Analysis for Fatigue Experiments, ASTM STP 588,* Am. Soc. Testing Mats., 1975. See also Little, R. E. and Jebe, E. H., *Statistical Design of Fatigue Experiments,* Applied Science Publishers, Ltd., London, 1975.

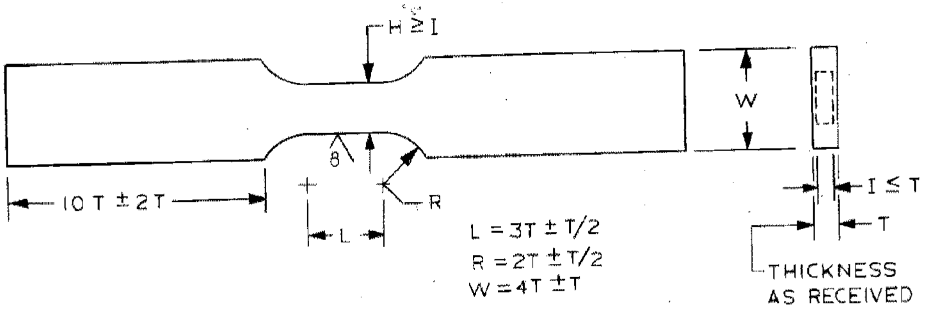
(5) Landgraf, R. W., Morrow, JoDean, and Endo, T., "Determination of the Cyclic Stress - Strain Curve," *Journal of Materials,* JMLSA, Vol 4, No. 1, March 1969, pp. 176–188.

(6) Metals Handbook, *Fractography and Atlas of Fractographs,* Am. Soc. Metals, Vol 9, Eighth Edition, 1974.



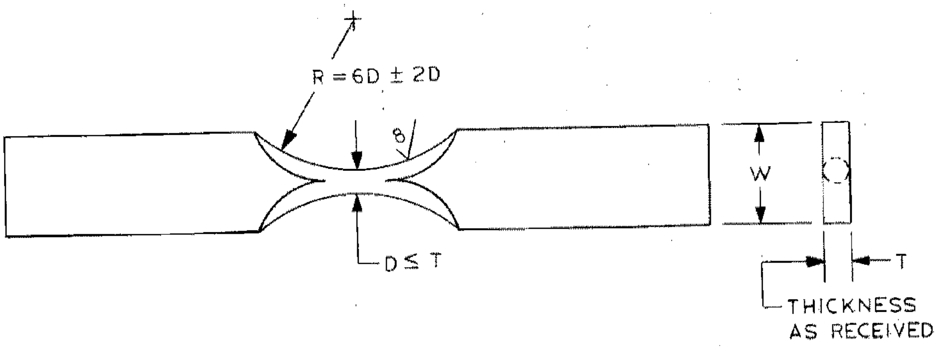
NOTE—Dimension *d* is recommended to be 6.35 mm (0.25 in.). See 7.1.

FIG. 1 Recommended Low-Cycle Fatigue Specimens.



Note—2.54 mm (0.1 in.) ≤ I ≤ T.

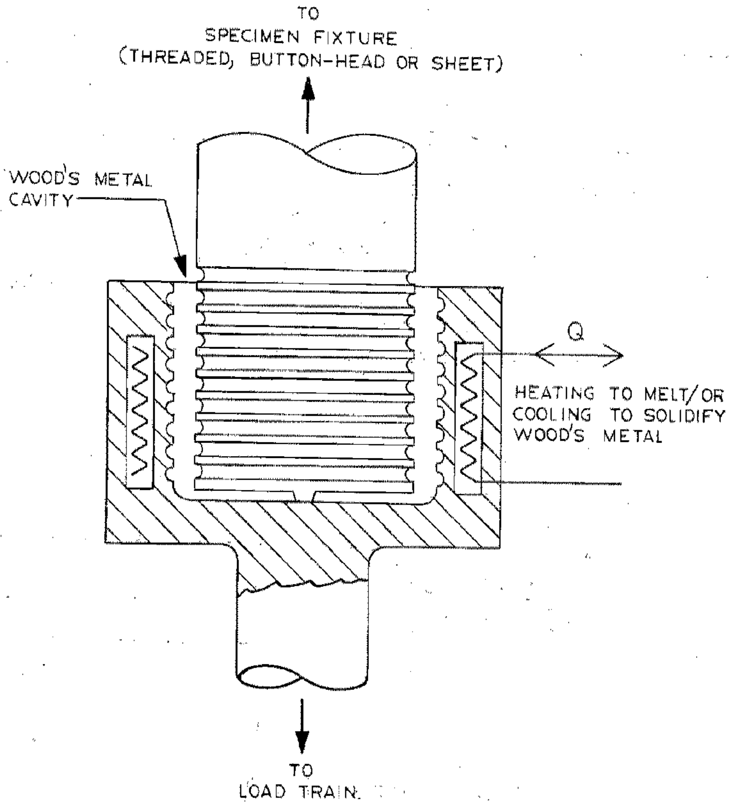
(*a*) Flat-Sheet Fatigue Specimen with Rectangular Cross Section.



Note—2.54 mm (0.1 in.) ≤ D.

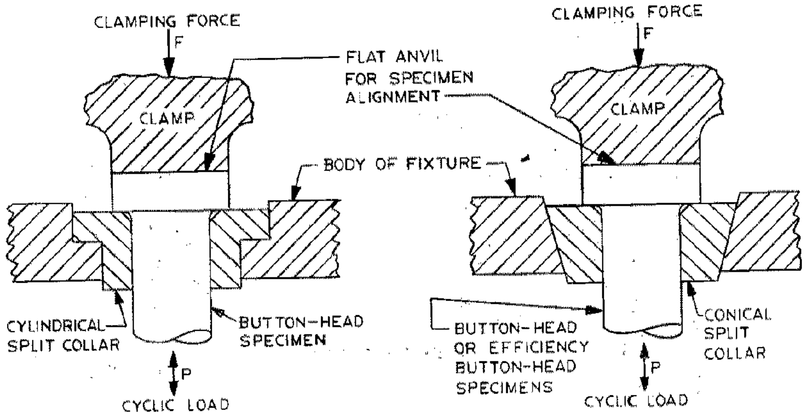
(*b*) Flat-Sheet Fatigue Specimen with Circular Cross Section.

FIG. 2 Sheet Fatigue Specimens—Alternative to Fig. 1 Specimens.

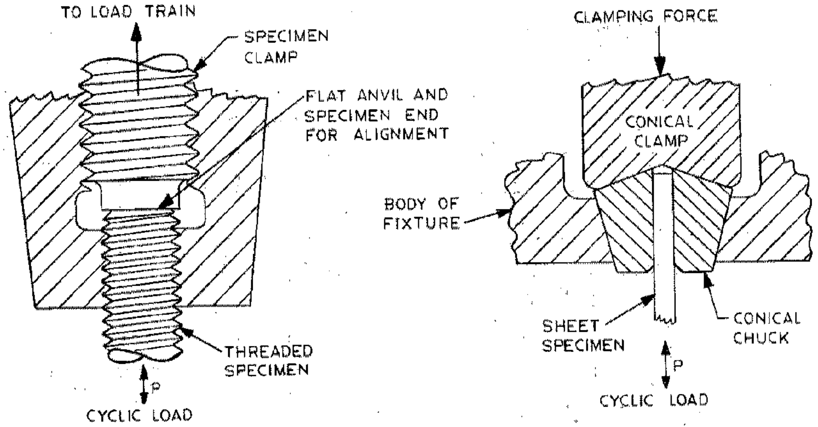


NOTE—Wood's metal pot is used to provide initially zero stress in the specimen during fixturing. This pot may be placed within a die-set to combine zero fixturing stress with rigid alignment.

FIG. 3 Schematic of Wood's Metal Pot Showing Principle of Operation.



(*a*) Button-Heat Fixture. (*b*) Button-Head or Efficiency Button-Head Fixture.



(*c*) Threaded Specimen Fixture. (*d*) Sheet Specimen Fixture.

NOTE—The clamping force should be greater than the cyclic load to avoid backlash within the specimen fixture.

FIG. 4 Schematic Examples of Fixturing Techniques for Various Specimen Designs.

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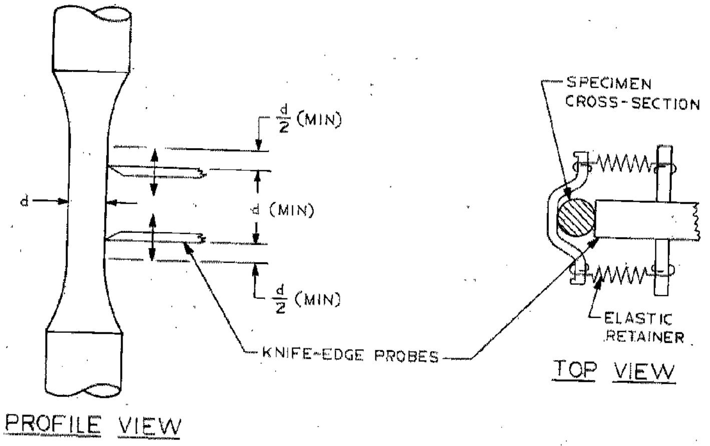


FIG. 5(*a*) Longitudinal Displacement Measurement for Uniform Gage Specimen of Fig. 1(*a*). (Probes may Be Attached to Either Transducer 5(*c*) or 5(*d*).)

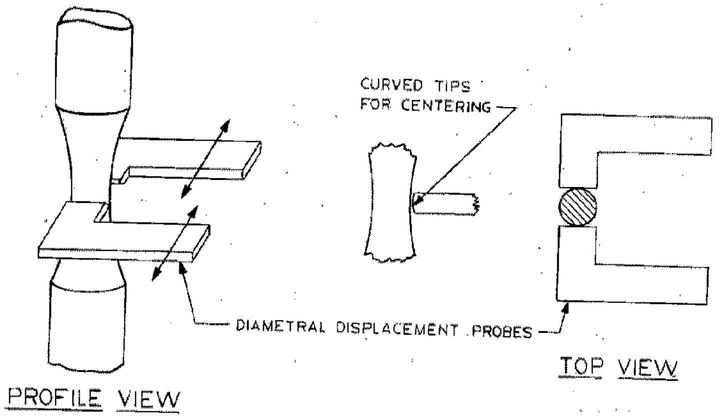


FIG. 5(*b*) Diametral Displacement Measurement for Hourglass Specimen of Fig. 1(*b*). (Probes may Be Attached to Either Transducer 5(*c*) or 5(*d*).)

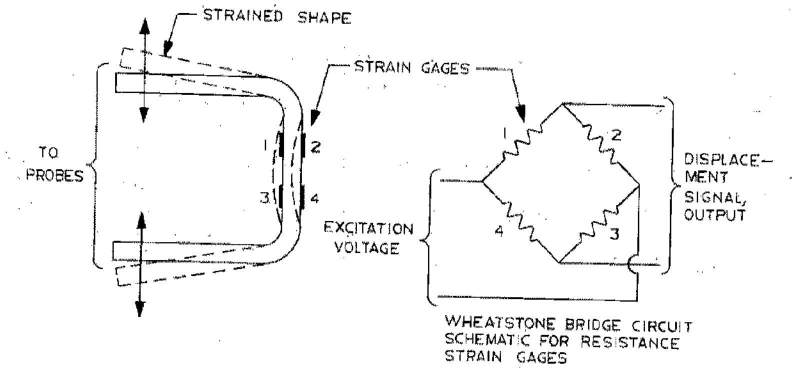


FIG. 5(*c*) Strain-Gage Displacement Transducer.

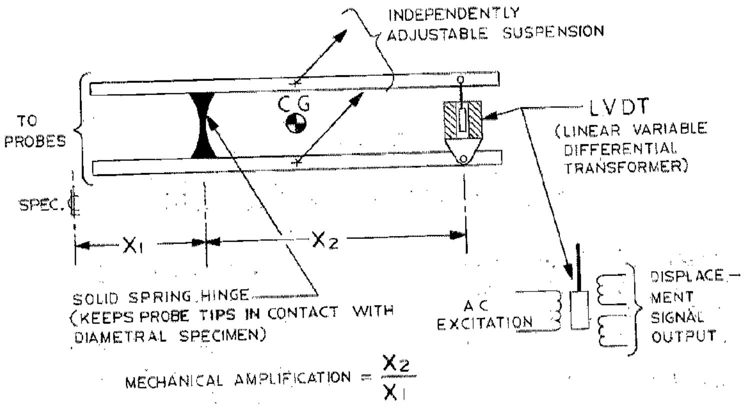


FIG. 5(*d*) LVDT Displacement Transducer.

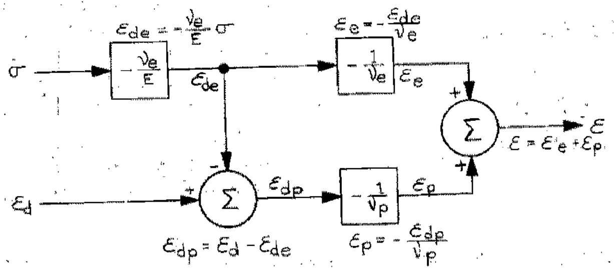
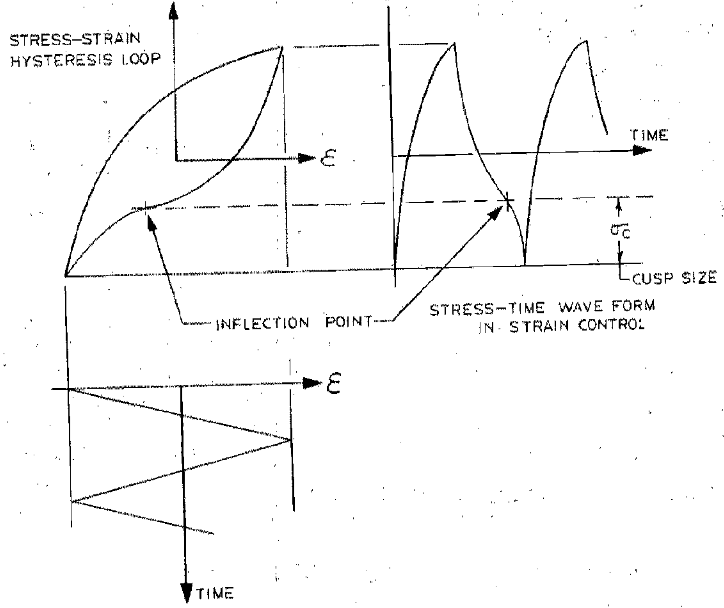


FIG. 6 Block Diagram of Strain Computer (See Appendix X2 for Discussion of Mathematical Relationships).



NOTE—The inflection point for a *stress-control* test may be similarly identified on the hysteresis loop and the *strain-time* diagram.

FIG. 7 Definition of "Cusp Size" for a Determination of Failure (See 8.9.3).

APPENDIXES

X1. FUNCTIONAL RELATIONSHIPS

X1.1 For many metals, the following empirical relationships (1) have been used for a convenient description of low-cycle fatigue data:

X1.1.1 *Cyclic Stress - Strain Behavior:*

$$\Delta\sigma/2 = K'(\Delta\epsilon_p/2)^{n'} \tag{1}$$

Note that Eq 1 may be expressed in terms of total strain range, $\Delta\epsilon$, by recognizing that $\Delta\epsilon = (\Delta\sigma/E) + \Delta\epsilon_p$.

X1.1.2 *Fatigue-Life Relationships:*

$$\Delta\sigma/2 = \sigma_f'(2N_f)^b \tag{2}$$

$$\Delta\epsilon_p/2 = \epsilon_f'(2N_f)^c \tag{3}$$

$$\Delta\epsilon/2 = [\sigma_f'/E](2N_f)^b + \epsilon_f'(2N_f)^c \tag{4}$$

where the variables are:

$\Delta\sigma$ = true stress range,
$\Delta\epsilon$ = true strain range,
$\Delta\epsilon_p$ = true plastic strain range,
N_f = cycles to failure, and
$2N_f$ = reversals to failure;

and the constants are:

n' = cyclic strain hardening exponent,
b = fatigue strength exponent,
c = fatigue ductility exponent,
K' = cyclic strength coefficient,
σ_f' = fatigue strength coefficient,
ϵ_f' = fatigue ductility coefficient, and
E = modulus of elasticity.

NOTE—The user is cautioned that Eqs 2 to 4 are not readily amenable to the use of conventional statistical analysis procedures.

X2. CONVERSION FROM DIAMETRAL STRAIN TO AXIAL STRAIN FOR ISOTROPIC MATERIALS

X2.1 Conversion of diametral strain to axial strain requires first the separation of the elastic and plastic components from the total strain by:

$$\begin{aligned} \epsilon &= \epsilon_e + \epsilon_p \\ \epsilon_d &= \epsilon_{de} + \epsilon_{dp} \end{aligned} \tag{1}$$

where:

e = elastic components,
p = plastic components,
d = diametral components, and
ϵ = total axial strain.

Axial and diametral components of strain are related through Poisson's ratio, ν, as:

$$\epsilon_e = -\epsilon_{de}/\nu_e \text{ and } \epsilon_p = -\epsilon_{dp}/\nu_p \tag{2}$$

The above expressions may be rearranged to yield:

$$\epsilon_{dp} = \epsilon_d - \epsilon_{de}$$

$$\epsilon = -\epsilon_{de}/\nu_e - (\epsilon_d - \epsilon_{de})/\nu_p \tag{3}$$

The diametral elastic strain, ϵ_{de}, is related to axial stress by means of Poisson's ratio and modulus,

$$\epsilon_{de} = -(\nu_e\sigma)/E$$

Thus:

$$\epsilon = \sigma/E - \epsilon_d/\nu_p - (\nu_e\sigma)/(\nu_p)E \tag{4}$$

Assuming that plastic deformation occurs under a constant volume condition:

$$\nu_p = 1/2$$

such that:

$$\epsilon = (\sigma/E)(1 - 2\nu_e) - 2\epsilon_d$$

In an experiment using a diametral strain gage and an axial load transducer, analogs of σ and ϵ_d are continuously available. Provided that elastic constants do not change with cyclic loading, the ratio ν_e/E can be found initially from the slope of the elastic portion of the σ versus ϵ_d curve and the modulus, E, may be determined in accordance with Method E 111.

X3. EXAMPLE OF MACHINING PROCEDURE

X3.1 While the following procedure was developed for machining high-strength materials with minimal attendant surface damage and alteration, it can be successfully applied to materials of lower strength. As a conservative general measure, this procedure is recommended unless: (*1*) the experimental objective is to evaluate another given surface condition or, (*2*) it is known that the material under evaluation is relatively insensitive to surface condition.

X3.2 **Procedure**

X3.2.1 In the final stages of machining, remove material in small amounts until 0.125 mm (0.005 in.) of excess material remains.

X3.2.2 Remove the next 0.1 mm (0.004 in.) of gage diameter by cylindrical grinding at a rate of no more than 0.005 mm (0.0002 in.)/pass.

NOTE X1—Some cast materials will not benefit from successive removal of material in small amounts, although this procedure is probably not to their detriment.

X3.2.3 Remove the final 0.025 mm (0.001 in.) by polishing (Note X2) longitudinally to impart a maximum of 0.2-μm (8-μin.) surface roughness.

NOTE X2—Extreme caution should be exercised in polishing to ensure that material is being properly removed rather than merely smeared to produce a smooth surface. This is a particular danger in soft materials wherein material can be smeared over tool marks, thereby creating a potentially undesirable influence on crack initiation during testing.

X3.2.4 After polishing (Note X2), all remaining grinding and polishing marks should be longitudinal. No circumferential machining should be evident when visualized at approximately 20× magnification under a light microscope.

X3.2.5 If specimen material is soft (for example, copper, aluminum, lead, etc.) at room temperature, final material removal can be performed by means of turning (rather than grinding) and subsequent polishing.

X3.2.6 Degrease the finished specimen.

X3.2.7 If heat treatment is necessary, conduct it before final machining or in such a manner as to avoid any surface damage; employ an inert protective atmosphere to eliminate surface oxidation.

X3.2.8 If surface observations are to be made, the test specimen may be electropolished in accordance with Method E 3.

X3.2.9 Imprint specimen numbers on both ends of the test section in regions of low stress, away from grip contact surfaces.

X4. ELEVATED-TEMPERATURE TESTING

X4.1 This Appendix is meant to provide guidance for conducting low-cycle fatigue tests at elevated temperatures where time-dependent strains can be present. Large segments of this recommended practice are already applicable to testing at these temperatures. The following paragraphs provide the necessary modifications to extend the practice for use in this regime. The numbers in parentheses refer to sections to which the paragraphs apply.

X4.1.1 Testing is permitted at temperatures and strain-rates where the magnitudes of time-dependent inelastic strains may be significant, relative to the magnitude of time-independent inelastic strains (1.3).

X4.1.2 No specific limitations are placed on the type of stress or strain cycling to which the test specimens are subjected. In particular, hold times on stress or strain are permitted (1.4).

X4.1.3 The presence of time-dependent inelastic strains during elevated temperature testing provides the opportunity to study the effects of these strains on fatigue life and on the cyclic stress-strain response of the material. Information about strain rate effects, relaxation behavior, and cyclic creep may also be available from these kinds of tests (3.3).

X4.1.4 Additional definitions will be found at the end of this appendix to cover elevated-temperature testing (4.1).

X4.1.5 The empirical relationships given in Appendix XI may not be valid when large time-dependent, inelastic strains are present. For this reason, all raw data should be reported to the extent possible. Data-reduction methods should be elaborated. If the data are also analyzed, details of the approach should be elaborated and information should be presented to permit analysis by other currently popular techniques (5.1).

X4.1.6 Satisfactory stress control cycling in the elevated temperature regime may require a greater degree of control on stress limits than specified in 6.2. Time-dependent effects can permit very large strains to be accumulated as a result of relatively slight errors in stress control. Consequently, special care should be exercised in this regime to avoid undesirable creep strain excursions. When, because of high cycle and long life considerations strain control testing is not practical, a pseudo strain control experiment under stress control is sometimes performed. In this case, it is desirable to monitor the strain during the test and to make modifications to the stress as necessary to remain within the required strain limits (6.2).

X4.1.7 For elevated-temperature testing it is usually necessary to provide some means for cooling the fixtures to prevent damage to other load-train components such as load cells. One method commonly used employs water-cooling coils attached to the fixtures or to other appropriate locations in the load train. Care must be taken to avoid affecting the load cell calibration or the load train alignment by the addition of cooling coils (6.3.2).

X4.1.8 Temperatures are permitted that may be high enough to produce significant time-dependent inelastic strains. Data should be presented in sufficient detail to facilitate their use to evaluate the utility of currently popular analysis techniques (8.1.1).

X4.1.9 When time-dependent effects are present, it may no longer be acceptable to control only the limits of the required stress or strain. Continuous control of the parameter of interest may be necessary to obtain the desired intra-cycle response. For example, if the load is controlled between total axial strain limits in this regime, a quite different material response will be produced than if the total axial strain is continuously controlled (8.3.1).

X4.1.10 Additional precautions should be observed when hold times are employed and time-dependent inelastic strains are present. For example, a hold on diametral strain will permit the total axial strain to change during each cycle and will not produce correct relaxation information (8.3.2).

X4.1.11 The additional complications of complex waveforms and time-dependent inelastic strains severely curtail the acceptability of limit control techniques. If the technique of limit control is used, the intra-cycle and inter-cycle variation of the parameter of interest should be monitored, and if necessary, periodic adjustments should be made to the testing machine to produce the desired response. Such changes should be reported (8.3.3).

X4.1.12 In addition to the preference for triangular waveforms for continuous cycling tests, trapezoidal waveforms with adjustable hold periods are needed for hold-time testing (8.4).

X4.1.13 It should be noted that if time-dependent effects are present, a reduced testing rate for recording purposes may produce significantly altered stress-strain response from that which would be produced at the normal testing rate. In addition, a possible effect of this periodic reduction of rate on specimen life should be considered (8.5.2, Note 11).

X4.1.14 No specific data analysis techniques are recommended for use when significant time-dependent inelastic strains are present (8.11).

X4.1.15 Dimensional instability of the specimen (unintentional changes in specimen geometry) may occur during the test as a result of time-dependent inelastic deformations. The post-mortem examination should include an evaluation of this occurence (8.11.3).

X4.1.16 Relaxation information should also be included for hold-time tests. This should include the values of the relaxed stress, the total amount of relaxation, and the change in the amount of inelastic strain during the hold period. Periodic stress-time recordings obtained with an expanded time scale may be necessary to obtain the desired resolution and to fully characterize the relaxation behavior (9.1.7.1).

X4.2 **Definitions**—In addition to the modification of certain sections as noted above, the following definitions will be used:

X4.2.1 *inelastic strain*, ϵ_{in}—that strain which is not elastic. For isothermal conditions ϵ_{in} is calculated by subtracting the elastic strain from the total strain.

X4.2.2 *hold period*, τ_h—a time interval within a cycle during which the stress or strain is held constant.

X4.2.3 *total cycle period*, τ_t—the time for the completion of one cycle.

Note—τ_t can be separated into hold and non-hold components, or as follows:

$$\tau_t = \Sigma\tau_h + \Sigma\tau_{nh} \tag{1}$$

where:

$\Sigma\tau_h$ = sum of all the hold portions of the cycle.

$\Sigma\tau_{nh}$ = sum of all the non-hold portions of the cycle.

τ_t is also equal to the reciprocal of the overall frequency when the frequency is held constant.

X4.2.4 *creep strain increment*, $\Delta\epsilon_c$—the change in the magnitude of the inelastic strain during a stress hold period.

X4.2.5 The following equations (refer to Fig. X1) are often used to define the instantaneous stress and strain relationships for many metals and alloys:

$$\epsilon = \epsilon_{in} + \epsilon_e, \text{ and} \qquad \epsilon_e = \frac{\sigma}{E^*}, \text{ (Note X1)} \tag{2}$$

and the change in strain from any point 1 to any other point 3 can be calculated as follows:

$$\epsilon_3 - \epsilon_1 = \left(\epsilon_{3in} + \frac{\sigma_3}{E^*}\right) - \left(\epsilon_{1in} + \frac{\sigma_1}{E^*}\right) \tag{3}$$

All strain points to the right of and all stress points above the origin are positive. The equation would then show an increase in inelastic strain from 1 to 3, or

$$\epsilon_{3in} - \epsilon_{1in} = \epsilon_3 - \epsilon_1 + \frac{\sigma_1}{E^*} - \frac{\sigma_3}{E^*} \tag{4}$$

Similarly, during the strain hold period, the change in the inelastic strain will be equal to the change in the stress divided by E^*, or

$$\epsilon_{3in} - \epsilon_{2in} = \frac{\sigma_2 - \sigma_3}{E^*} \tag{5}$$

Note X1—E^* represents a material variable that may be a function of environment and test conditions. It may also vary during a test as a result of metallurgical or physical changes in the specimen. In many instances, however, it is practically a constant quantity and is used rather extensively, in isothermal, constant-rate testing, in the analysis of hysteresis loops. A value for E^* can best be determined by cycling the specimen prior to the test at stress levels below the elastic limit.

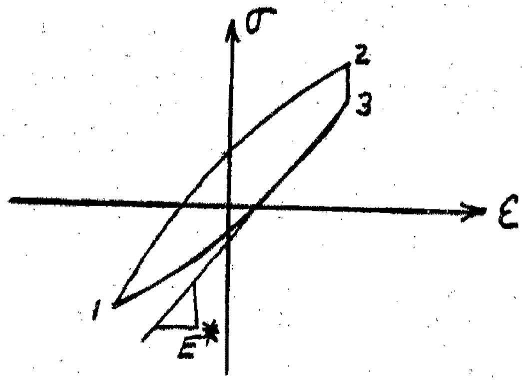


FIG. X1 Analysis of a Total Strain Versus Stress Hysteresis Loop Containing a Hold Period

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* * *

Document Name: ASTM E695: Standard Method of Measuring Relative Resistance of Wall, Floor and Roof Construction to Impact Loading

CFR Section(s): 24 CFR 200.946(a)(1)(viii)

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 695 – 79 (Reapproved 1997)$^{\epsilon 1}$

An American National Standard

Standard Method of Measuring Relative Resistance of Wall, Floor, and Roof Construction to Impact Loading[1]

This standard is issued under the fixed designation E 695; 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.

ϵ^1 NOTE—Keywords were added editorially in March 1997.

1. Scope

1.1 This method covers the measurement of the relative resistance of wall, floor, and roof construction to impact loading. The test is not applicable to doors.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1517 Definitions of Terms Relating to Leather[2]

E 73 Practice for Static Load Testing of Truss Assemblies[3]

E 575 Practice for Reporting Data from Structural Tests of Building Constructions, Elements, Connections, and Assemblies[3]

E 661 Test Method for Performance of Wood and Wood-Based Floor and Roof Sheathing Under Concentrated Static and Impact Loads[3]

2.2 *Other Standards:*

ANSI Z 299.2 Voluntary Industry Performance Standards for Pressure and Velocity of Shotshell Ammunition for the Use of Commercial Manufacturers[4]

Federal Specification V-T-291E (1) Thread, Linen[5]

3. Significance and Use

3.1 The procedures outlined will provide data that can be used to evaluate the relative performance of wall, floor, and roof constructions under conditions representative of those sustained in actual service when subjected to impact by a heavy blunt object. See Test Method E 661 for evaluation of floor and roof sheathing and Practice E 73 for evaluation of roof trusses.

3.2 The method is intended to be applied to relatively light construction, including but not limited to wood floor and roof systems, partitions framed with wood or steel studs, steel floor or roof decking systems, steel siding and wall panels, or thin concrete and masonry walls or slabs and similar assemblies.

4. Summary of Method

4.1 Specimens of wall, floor, and roof construction are subjected to the impact force of a standard impact instrument. Wall sections are tested in the vertical position. Floor and roof sections are tested only in the horizontal position. Because of the inherent differences in the method of applying load, measurements obtained from tests in a horizontal mode are not comparable to measurements obtained from tests in the vertical mode.

5. Apparatus for Floor and Roof Systems, Specimen Horizontal (see Fig. 1)

5.1 *Supports*, steel rollers, two, on a rigid base.

5.2 *Impact Instrument*, made with a lead shot-filled leather bag as specified in 5.2.1-5.2.6.

5.2.1 *Leather*—The leather used in construction of the bag should be harness leather (Note 1), oak tanned (Note 1) from packer hides (Note 1) or latigo leather (Note 1), alum and vegetable tanned, or both. Leather thickness shall be expressed in ounces (Note 1) (1 oz = 0.4 mm (1/64 in.)).

NOTE 1—See Definitions D 1517.

5.2.2 *Thread*—Thread used in fabrication of the bag shall be linen thread of four or more plys, meeting the requirements for Type B, Class 1 or 2, of Federal Specification V-T-291E (*1*).

5.2.3 *Fabrication*—The side of the bag shall be 710 mm (28 in.) high by 735 mm (29 in.) wide of 8-oz leather 3 mm (1/8 in.) thick. The vertical edges shall be sewed together flesh side out and the seam shall be reinforced with a piece of 8-oz leather overlapping 10 mm (3/8 in.) each side. The side shall then be turned hair side out and sewed to the bottom. The base (bottom disk) shall be 230 mm (9 in.) in diameter of 12-oz leather 5 mm (3/16 in.) thick. The seam attaching the wall to the base shall be

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[1] This method is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.11 on Horizontal and Vertical Structures/Structural Performance of Completed Structures.

Current edition approved Oct. 29, 1979. Published February 1980.

[2] *Annual Book of ASTM Standards*, Vol 15.04.

[3] *Annual Book of ASTM Standards*, Vol 04.11.

[4] Available from American National Standards Institute, 11 West 42nd St, 13th Floor, New York, NY 10036.

[5] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

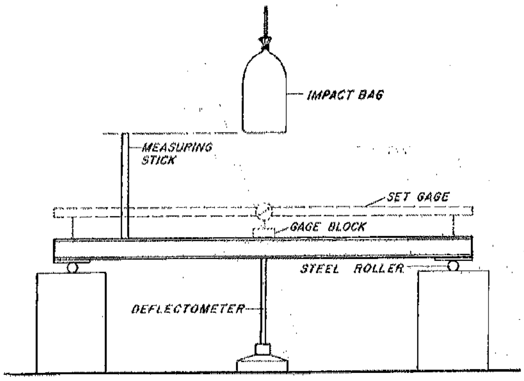

FIG. 1 Impact Load Test (Specimen Horizontal)

6 mm (¼ in.) from the edge of the base. Two rows of stitching shall be used for the vertical wall seam and the seam attaching the wall to the base.

5.2.4 *Hoisting Strap*—The strap to hoist the bag shall be made from 8-oz leather 3 mm (⅛ in.) thick by 15 mm (⅝ in.) wide by 610 mm (24 in.) long. The strap shall be passed through holes, diametrically opposite, in the side walls 40 mm (1½ in.) from the top of the wall. These holes shall be reinforced with pieces of 8-oz leather and 75 mm (3 in.) square. The leather strap shall be passed twice through a 50-mm (2-in.) diameter lifting ring and the ends fastened by sewing, riveting, or by use of a buckle. To avoid excessive stretching of the leather wall or failure of the vertical seam, a sleeve, made from 12-oz leather, of the same type as the base of the bag, shall be fitted to slip tightly around the lower portion of the bag. This sleeve should be 250 mm (9⅝ in.) high.

5.2.5 *Lead Shot*—The bag shall be loosely filled with No. 7½ (2.4 mm (0.095 in.) in diameter) chilled lead shot, in conformance with ANSI Z 299.2. Two layers of 75-mm (3-in.) thick foam rubber or similar padding shall be placed over the lead shot to prevent spillage during testing.

5.2.6 The total mass of the bag, including shot, shall be adjusted to the desired level with an accuracy of ±1 %. The mass of the bag may be adjusted to any specified mass, depending upon the information desired.

5.3 *Measuring Sticks*—A stick, laid off in 150-mm (6-in.) increments, or a series of sticks the lengths of which are multiples of 150 mm (6 in.), to measure the height of drop accurately. A graduated sliding pointer, a standard metal tape measure, or any similar device that can accurately measure the height of drop may be substituted.

5.4 *Deflectometer*, or other suitable deflectometer equipment, consisting of a metal tube having a base at the lower end and a clamp at the upper end which supports, by friction, a light metal rod. The rod shall be movable inside the tube and shall be graduated to 0.25-mm (0.01-in.) divisions.

5.5 *Set Gage*, consisting of a light, rigid frame having two legs at one end and one leg at the other end, with the distance between the legs equal to the span of the specimen. A dial micrometer graduated to 0.025-mm (0.001-in.) divisions shall be attached to the frame at midlength.

5.6 *Gage Blocks*, 300 by 300 mm (12 by 12 in.) in area, and constructed of metal or other hard surface material.

5.7 *Hold-Downs*—Clamps or other restraining devices at the specimen ends to minimize translation.

6. Apparatus for Wall Systems, Specimen Vertical (see Fig. 2)

6.1 *Steel Channels*, for support of the specimen at top and bottom.

6.2 *Rollers*, cylindrical rollers and two supporting rollers.

6.3 Impact bag, measuring sticks, deflectometer, set gage, and gage blocks conforming to the requirements specified in 5.2-5.7.

6.4 *Rigid Supporting Frame*, to which the supporting channels and deflection gage are attached.

7. Test Specimen

7.1 *Size*—The specimens shall be representative of the actual construction as to material, method of assembly, and workmanship.

7.2 *Length or Height*—The length or height of specimen for each element shall be chosen to conform approximately to the length or height of that element in actual size.

7.3 *Width*—The width of specimen shall be chosen, insofar as feasible, to include several of the principal load-carrying members to ensure that the behavior under load will simulate that anticipated under service conditions. The actual width of specimens shall be a whole number multiplied by the spacing of the principal load-carrying members, except for prefabricated panels for which the actual width shall be the width of panel used. If the structural properties of a particular construction are to be compared with another construction, there should not be a great difference in the actual widths of the specimens.

7.4 *Age*—Constructions such as concrete and masonry (brick, structural clay tile, concrete block) for which the structural properties depend upon the age of the specimen, shall be tested not less than 25 days nor more than 56 days after fabrication except in special instances such as the case of existing panels. This age requirement applies also to plastered and stuccoed constructions. Other assemblies affected by moisture shall be conditioned to constant weight or moisture content, or for at least 2 weeks at 20 ± 3°C (68 ± 6°F) and 65 ± 5 % relative humidity.

7.5 *Number*—Tests shall be made on a minimum of three like specimens. However, more tests may be necessary depending upon information and accuracy desired.

8. Procedure

8.1 For symmetrical walls apply the impact load to the outside face from at least one of the specimens, and to the inside face of the other two specimens. For asymmetrical walls, test both sides an equal number of times. This will require a minimum of four test specimens. Exception: only one side need be tested at the option of the client and laboratory depending upon information required. The report shall record which side or sides of the specimen that is tested. For floor and roof assemblies apply the impact loads only to the upper finish-floor face of the specimen.

Note 2—Any criteria for pass/fail or for stopping the test should be agreed upon between the sponsor and testing agency prior to testing. Some possible points include: the ability to sustain a specified static load after

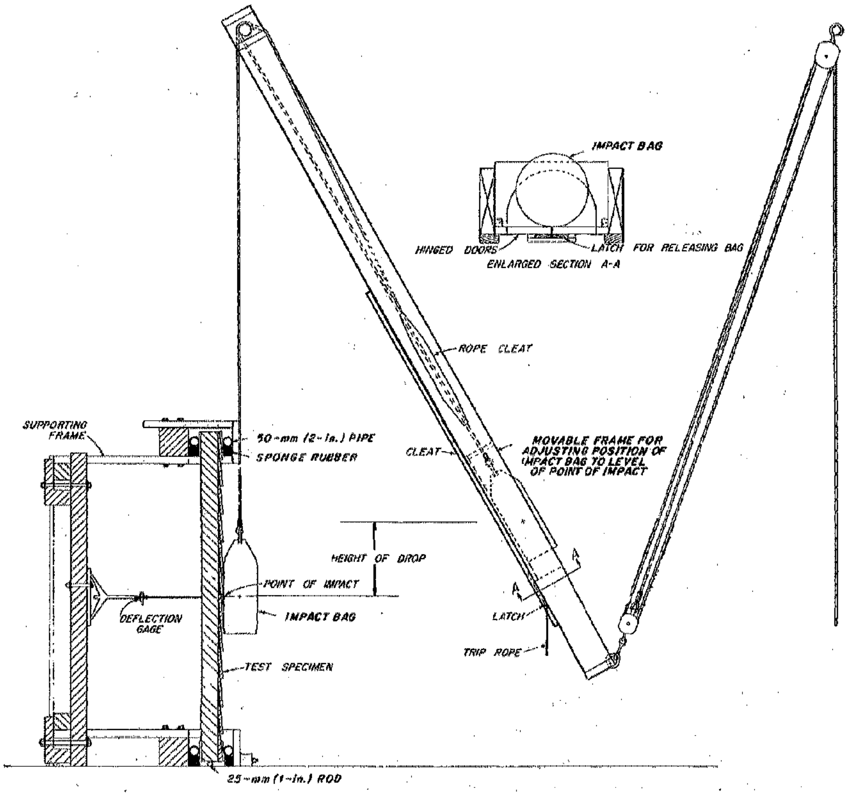

FIG. 2 Impact Load Test (Specimen Vertical)

the impact drop; limitation on the instantaneous deflection or residual set; penetration of panel; destruction of panel (to be defined); or simply a specified height of drop or number of drops per test from a specific height.

8.2 *Point of Impact*—If the construction has structural members, that is, studs or joists, test each specimen so that at least one such structural member will be struck by the impact bag. Test each specimen also so that the impact bag will strike the facing midway between two members.

Note 3—As written, this method may not necessarily define or test the most vulnerable part of a panel specimen. Additional tests may be required to locate such an area if this information is desired.

8.3 *Loading-Floor and Roof Assemblies-Specimen Horizontal*—Test the specimen as a simple beam on a span nominally 150 mm (6 in.) less than the specimen length. The two supports for the specimens shall prevent longitudinal restraint and shall provide bearing for the entire width of the specimen. Secure the ends of the panel by hold-downs to minimize specimen bounce. Take care to assure that the hold-downs do not affect deflection of the specimen. Apply an impact load to the upper face of the specimen by dropping the bag beginning with a height of 150 mm (6 in.) and increasing the height in 150-mm increments. Record set and instantaneous deflection measurements for each drop. For the first drop, measure the height of the bag from the upper face of the specimen at a point directly beneath the bag, and for subsequent drops, from a taut cord in contact with the upper face directly above the supports.

8.4 *Loading-Wall Assemblies Specimen Vertical*—Position the specimen on cylindrical rollers to prevent transverse restraint. The axes of the rollers shall be parallel to the faces of the specimens. The two supporting rollers shall be in contact with the vertical surface of the rigid frame and each roller shall rest horizontally on sponge rubber about 15 mm (½ in.) thick to prevent longitudinal restraint. Support the bag as a pendulum in the frame as shown in Fig. 2. Take care to assure that the hold-downs do not affect deflection of the specimen. Apply an impact load to the middle of the outer face of the specimens by releasing the bag beginning with a height of 150 mm (6 in.) and increasing the height in 150-mm increments. The maximum useful height of drop will occur when the pendulum or frame is perpendicular to the specimen. Measure the height of drop from the point of impact of the center of gravity of the bag as it strikes the specimen to this same point when the bag is in the raised position. Release the bag by smoothly and swiftly opening the hinged doors, causing it to swing as a true pendulum thus eliminating wobbling.

8.5 *Instantaneous Deflection*—Use the deflectometer (see 5.4) to measure the instantaneous deflection of the specimen. Prior to loading, hold the light metal rod in contact with the middle of the lower face of the specimen by the clamp. When the specimen deflects under the impact load, the rod is held in its lowest position by the friction clamp. Report readings to the nearest 0.25 mm (0.01 in.).

8.6 *Set*—To measure the set, place the set gage on the upper face of the specimen. Take readings by placing the set gage on the specimen with the legs at the supports and the spindle of the dial micrometer in contact with the middle of the specimen, and reading the micrometer. If the set exceeds the range of the micrometer, place gage blocks between the specimen and the spindle of the micrometer to the nearest 0.025 mm (0.001 in.).

9. Recordings

9.1 *Deflection and Set*—For each height of drop calculate the deflection between the reading of the deflectometer and the initial reading. Similarly, calculate the set as the difference between the reading of the set gage and the initial reading. Record the maximum height of drop.

10. Report

10.1 The report shall follow the format of Practice E 575. In addition, the report shall also include the following:

10.2 Description of test assembly, including:

10.2.1 Size of test specimen,

10.2.2 Details of structural design, including where applicable or required, the design stresses, design loads and safety factors of all structural members in test assembly,

10.2.3 Plan, evaluation, principal cross section, plus other sections as needed for clarity, and

10.2.4 Details of attachment of test panel in frame.

10.3 Summarize results.

10.4 It is essential that drawings required by 3.1.6 of Practice E 575 be provided.

11. Precision and Bias

11.1 Neither the within-laboratory nor the between-laboratory precision and bias of the impact load procedures recommended have been established, because any test data developed are usually of a proprietary nature and unavailable. Furthermore, the test method is expected to be used for prototype testing and not for routine quality control. This means few specimens of a kind would be tested, making any analysis unjustifiable.

12. Keywords

12.1 floor systems; impact loading; roof systems; wall systems

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Document Name: ASTM E711: Standard Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter

CFR Section(s): 40 CFR 63, Subpart DDDDD, Table 6

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 711 – 87 (Reapproved 1992)

Standard Test Method for Gross Calorific Value of Refuse-Derived Fuel by the Bomb Calorimeter[1]

This standard is issued under the fixed designation E 711; 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 test method covers the determination of the gross calorific value of a prepared analysis sample of solid forms of refuse-derived fuel (RDF) by the bomb calorimeter method.

1.2 *This standard does not purport to address all of the safety problems, 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 cautionary and precautionary statements see 6.10 and Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 3177 Test Method for Total Sulfur in the Analysis Sample of Coal and Coke[3]

E 1 Specification for ASTM Thermometers[4]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[5]

E 775 Test Methods for Total Sulfur in the Analysis Sample of Refuse-Derived Fuel[6]

E 790 Test Method for Residual Moisture in a Refuse-Derived Fuel Analysis Sample[6]

E 829 Practice for Preparing Refuse-Derived Fuels (RDF) Laboratory Samples for Analysis[6]

3. Terminology

3.1 *Definitions:*

3.1.1 *calorific value*—the heat of combustion of a unit quantity of a substance. It may be expressed in joules per gram (J/g), British thermal units per pound (Btu/lb), or calories per gram (cal/g) when required.

NOTE 1—The unit equivalents are as follows:
1 Btu (International Table) = 1055.06 absolute joules
1 Calorie (International Table) = 4.1868 absolute joules
1 Btu/lb = 2.326 J/g
1.8 Btu/lb = 1.0 cal/g

3.1.2 *gross calorific value*—the heat produced by combustion of a unit quantity of solid fuel, at constant volume, in an oxygen bomb calorimeter under specified conditions such that all water in the products remains in liquid form.

3.1.3 *net calorific value*—a lower value calculated from the gross calorific value. It is equivalent to the heat produced by combustion of a unit quantity of solid fuel at a constant pressure of one atmosphere, under the assumption that all water in the products remains in the form of vapor.

3.2 *Descriptions of Terms Specific to This Method:*

3.2.1 *calorimeter*—describes the bomb, the vessel with stirrer, and the water in which the bomb is immersed.

3.2.2 *energy equivalent*—the energy required to raise the temperature (Note 2) of the calorimeter system 1°C (or 1°F) per gram of sample. This is the number that is multiplied by the corrected temperature rise in degrees and divided by the sample weight in grams to give the gross calorific value after thermochemical corrections have been applied.

NOTE 2—Temperature change is measured in thermal units. Temperature changes may also be recorded in electromotive force, ohms, or other units when other types of temperature sensors are used. Consistent units must be used in both the standardization and actual calorific determination. Time is expressed in minutes. Weights are measured in grams.

3.2.3 *refuse-derived fuels*—solid forms of refuse-derived fuels from which appropriate analytical samples may be prepared are defined as follows in *ASTM STP 832*:[7]

RDF-1—Wastes used as a fuel in as-discarded form with only bulky wastes removed.

RDF-2—Wastes processed to coarse particle size with or without ferrous metal separation.

RDF-3—Combustible waste fraction processed to particle sizes, 95 % passing 2-in. square screening.

RDF-4—Combustible waste fraction processed into powder form, 95 % passing 10-mesh screening.

RDF-5—Combustible waste fraction densified (compressed) into the form of pellets, slugs, cubettes, or briquettes.

4. Summary of Test Method

4.1 Calorific value is determined in this method by burning a weighed analysis sample in an oxygen bomb calorimeter under controlled conditions. The calorific value is computed from temperature observations made before and after combustion, taking proper allowance for thermometer and thermochemical corrections. Either isothermal or adiabatic calorimeter jackets may be used.

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[1] This test method is under the jurisdiction of ASTM Committee D-34 on Waste Management and is the direct responsibility of Subcommittee D34.08 on Thermal Treatment.

Current edition approved Aug. 28, 1987. Published October 1987.

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

[3] *Annual Book of ASTM Standards*, Vol 05.05.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 15.05.

[6] *Annual Book of ASTM Standards*, Vol 11.04.

[7] *Thesaurus on Resource Recovery Terminology, ASTM STP 832*, ASTM, 1983, p. 72.

5. Significance and Use

5.1 The calorific value, or heat of combustion, is a measure of the energy available from a fuel. Knowledge of this value is essential in assessing the commercial worth of the fuel and to provide the basis of contract between producer and user.

6. Apparatus

6.1 *Test Room*—The apparatus should be operated in a room or area free of drafts that can be kept at a reasonably uniform temperature and humidity for the time required for the determination. The apparatus should be shielded from direct sunlight and radiation from other sources. Controlled room temperature and humidity are desirable.

6.2 *Oxygen Bomb*, constructed of materials that are not affected by the combustion process or products sufficiently to introduce measurable heat input or alteration of end products. If the bomb is lined with platinum or gold, all openings shall be sealed to prevent combustion products from reaching the base metal. The bomb shall be designed so that all liquid combustion products can be completely recovered by washing the inner surfaces. There shall be no gas leakage during a test. The bomb shall be capable of withstanding a hydrostatic pressure test to 21 MPa (3000 psig) at room temperature without stressing any part beyond its elastic limit.

6.3 *Calorimeter*, made of metal (preferably copper or brass) with a tarnish-resistant coating and with all outer surfaces highly polished. Its size shall be such that the bomb will be completely immersed in water when the calorimeter is assembled. It shall have a device for stirring the water thoroughly and at a uniform rate, but with minimum heat input. Continuous stirring for 10 min shall not raise the calorimeter temperature more than 0.01°C (0.02°F) starting with identical temperatures in the calorimeter, room, and jacket. The immersed portion of the stirrer shall be coupled to the outside through a material of low heat conductivity.

6.4 *Jacket*—The calorimeter shall be completely enclosed within a stirred water jacket and supported so that its sides, top, and bottom are approximately 10 mm from the jacket walls. The jacket may be arranged so as to remain at constant temperature or with provisions for rapidly adjusting the jacket temperature to equal that of the calorimeter for adiabatic operation. It shall be constructed so that any water evaporating from the jacket will not condense on the calorimeter.

6.5 *Thermometers*—Temperatures in the calorimeter and jacket shall be measured with the following thermometers or combinations thereof:

6.5.1 *Mercury-in-Glass Thermometers*, conforming to the requirements for Thermometers 116°C or 117°C (56°F or 57°F) as prescribed in Specification E 1. Other thermometers of equal or better accuracy are satisfactory. These thermometers shall be tested for accuracy against a known standard (preferably by the National Bureau of Standards) at intervals no greater than 2.0°C (3.6°F) over the entire graduated scale. The maximum difference in correction between any two test points shall not be more than 0.02°C (0.04°F).

6.5.2 *Beckmann Differential Thermometer*, having a range of approximately 6°C in 0.01°C subdivisions reading upward and conforming to the requirements for Thermometer 115°C, as prescribed in Specification E 1. Each of these thermometers shall be tested for accuracy against a known standard at intervals no larger than 1°C over the entire graduated scale. The maximum difference between any two test points shall not be more than 0.02°C.

6.5.3 *Calorimetric-Type Platinum Resistance Thermometer, 25-*, tested for accuracy against a known standard.

6.5.4 *Other Thermometers*—A high precision electronic thermometer employing balanced thermistors or a quartz thermometer may be used, provided the temperature rise indication is accurate within ±0.003°C per 1°C rise.

6.6 *Thermometer Accessories*—A magnifier is required for reading mercury-in-glass thermometers to one tenth of the smallest scale division. This shall have a lens and holder designed so as to introduce no significant errors due to parallax. A Wheatstone bridge and galvanometer capable of measuring resistance to 0.0001 Ω are necessary for use with resistance thermometers.

6.7 *Sample Holder*—Samples shall be burned in an open crucible of platinum, quartz, or acceptable base-metal alloy. Base-metal alloy crucibles are acceptable if after a few preliminary firings the weight does not change significantly between tasks.

6.8 *Firing Wire* shall be 100 mm of No. 34 B & S nickel-chromium alloy wire or 100 mm of No. 34 B & S iron wire. Equivalent platinum or palladium wire may be used provided constant ignition energy is supplied, or measured, and appropriate corrections made.

6.9 *Firing Circuit*—A 6 to 16-V alternating or direct current is required for ignition purposes with an ammeter or pilot light in the circuit to indicate when current is flowing. A stepdown transformer connected to an alternating current lighting circuit or batteries may be used.

6.10 **CAUTION:** The ignition circuit switch shall be of momentary double-contact type, normally open, except when held closed by the operator. The switch should be depressed only long enough to fire the bomb.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall 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, where such specifications are available.[8] 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water, Type III, conforming to Specification D 1193.

7.3 *Benzoic Acid, Standard (C_6H_5COOH)*—Use National Bureau of Standards SRM (Standard Reference Material) benzoic acid. The crystals shall be pelletized before use. Commercially prepared pellets may be used provided they are made from National Bureau of Standards benzoic acid.

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[8] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Analar Standards for Laboratory U.K. Chemicals," BDH Ltd., Poole, Dorset, and the "United States Pharmacopeia."

The value of heat of combustion of benzoic acid, for use in the calibration calculations, shall be in accordance with the value listed in the National Bureau of Standards certificate issued with the standard.

7.4 *Methyl Orange, Methyl Red, or Methyl Purple Indicator* may be used to titrate the acid formed in the combustion. The indicator selected shall be used consistently in both calibrations and calorific determinations.

7.5 *Oxygen*, free of combustible matter. Oxygen manufactured from liquid air, guaranteed to be greater than 99.5 % pure, will meet this requirement. Oxygen made by the electrolytic process may contain a small amount of hydrogen rendering it unfit without purification.

7.6 *Sodium Carbonate, Standard Solution* (0.34 *N*)—One millilitre of this solution should be equivalent to 20.0 J in the nitric acid (HNO_3) titration. Dissolve 18.02 g of anhydrous sodium carbonate (Na_2CO_3) in water and dilute to 1 L. The Na_2CO_3 should be previously dried for 24 h at 105°C. The buret used for the HNO_3 titration shall be of such accuracy that estimations to 0.1 mL can be made. A more dilute standard solution may be used for higher sensitivity.

8. Precautions

8.1 Due to the origins of RDF in municipal waste, common sense dictates that some precautions should be observed when conducting tests on the samples. Recommended hygienic practices include use of gloves when handling RDF and washing hands before eating or smoking.

8.2 The following precautions are recommended for safe calorimeter operation:

8.2.1 The weight of solid fuel sample and the pressure of the oxygen admitted to the bomb must not exceed the bomb manufacturer's recommendations.

8.2.2 Bomb parts should be inspected carefully after each use. Threads on the main closure should be checked frequently for wear. The bomb should be returned to the manufacturer occasionally for inspection and possibly proof of firing.

8.2.3 The oxygen supply cylinder should be equipped with an approved type of safety device, such as a reducing valve, in addition to the needle valve and pressure gage used in regulating the oxygen feed to the bomb. Valves, gages, and gaskets must meet industry safety codes. Suitable reducing valves and adaptors for 2 to 3.5-MPa (300 to 500-psig) discharge pressure are obtainable from commercial sources of compressed gas equipment. The pressure gage shall be checked periodically for accuracy.

8.2.4 During ignition of a sample, the operator shall not permit any portion of his body to extend over the calorimeter.

9. Sampling[9]

9.1 RDF products are frequently nonhomogeneous. For this reason significant care should be exercised to obtain a representative laboratory sample for the RDF lot to be characterized.

9.2 The sampling method for this procedure should be based on agreement between the involved parties.

9.3 The laboratory sample must be air-dried and particle size reduced to pass a 0.5-mm screen as described in Practice E 829.

10. Standardization

10.1 Determine the energy equivalent of the calorimeter as the average of a series of ten individual runs, made over a period of not less than 3 days or more than 5 days. To be acceptable, the standard deviation of the series shall be 6.9 kJ/°C (6.5 Btu/°C) or less (see Appendix X1, Table X1). For this purpose, any individual run may be discarded only if there is evidence indicating incomplete combustion. If this limit is not met, repeat the entire series until a series is obtained with a standard deviation below the acceptable limit.

10.2 The weights of the pellets of benzoic acid in each series should be regulated to yield the same temperature rise as that obtained with the various samples tested in the individual laboratories. The usual range of weight is 0.9 to 1.3 g. Make each determination in accordance with the procedure described in Section 11, and compute the corrected temperature rise, *T*, as described in 12.1. Determine the corrections for HNO_3 and firing wire as described in 12.2 and substitute into the following equation:

$$E = [(H)(g) + e_1 + e_3 + e_4] \times t$$

where:

E = energy equivalent, J/°C,
H = heat of combustion of benzoic acid, as stated in the National Bureau of Standards certificate, J/g,
g = weight of benzoic acid, g,
t = corrected temperature rise, °C,
e_1 = titration correction, J,
e_3 = fuse wire correction, J, and
e_4 = correction for ignition energy if measured and corrected for, J.

10.3 Standardization tests should be repeated after changing any part of the calorimeter and occasionally as a check on both calorimeter and operating technique.

11. Procedure

11.1 *Weight of Sample*—Thoroughly mix the analysis sample of solid fuel in the sample bottle, taking care that the heavies and lights (fluff) are distributed in the sample (Note 3). Carefully weigh approximately 1 g of the sample directly into the crucible in which it is to be burned or into a tared weighing scoop from which the sample is transferred to the crucible. Weigh the sample to the nearest 0.1 mg. Some form of compaction may be necessary to ensure satisfactory ignition and complete combustion.

NOTE 3—In the event segregation of the heavies and lights cannot be avoided, attempt to remove sample from the bottle in such a way that a representative sample is transferred.

NOTE 4—Perform the residual moisture determination of the sample simultaneously using Test Method E 790.

11.2 *Water in Bomb*—Add 1.0 mL of water to the bomb by a pipet. Before adding this water, rinse the bomb, and drain the excess water, and leave undried.

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[9] ASTM Subcommittee E38.01 is currently in the process of developing procedures for sampling RDF-3 and the preparation of an analysis sample. The chairman of E38.01 should be contacted for details.

11.3 *Firing Wire*—Connect a measured length of firing wire to the ignition terminals with enough slack to allow the firing wire to maintain contact with the sample.

11.4 *Oxygen*—Charge the bomb with oxygen to a consistent pressure between 20 and 30 atm (2.03 and 3.04 MPa). This pressure must remain the same for each calibration and for each calorific determination. If, by accident, the oxygen introduced into the bomb should exceed the specified pressure, do not proceed with the combustion. Detach the filling connection and exhaust the bomb in the usual manner. Discard this sample.

11.5 *Calorimeter Water*—It is recommended that calorimeter water temperature be adjusted before weighing as follows:

11.5.1 *Isothermal Jacket Method,* 1.6 to 2.0°C (3.0 to 3.5°F) below jacket temperature (Note 4).

11.5.2 *Adiabatic Jacket Method,* 1.0 to 1.4°C (2.0 to 2.5°F) below room temperature.

NOTE 5—This initial adjustment will ensure a final temperature slightly above that of the jacket for calorimeters having an energy equivalent of approximately 10 200 J/K (2450 cal/°C). Some operators prefer a lower initial temperature so that the final temperature is slightly below that of the jacket. This procedure is acceptable, provided it is used in all tests, including standardization. Use the same amount (±0.5 g) of water in the calorimeter vessel for each test and for calibration. The amount of water (2000 g is usual) can be most satisfactorily determined by weighing the calorimeter vessel and water together on a balance. The water may be measured volumetrically if it is always measured at the same temperature. Tap water may be satisfactory for use in calorimeter bucket.

11.6 *Observations, Isothermal Jacket Method*—Assemble the calorimeter in the jacket and start the stirrer. Allow 5 min for attainment of equilibrium; then record the calorimeter temperatures (Note 6) at 1-min intervals for 5 min. Fire the charge at the start of the sixth minute and record the time and temperature, T^a. Add to this temperature 60 % of the expected temperature rise, and record the time at which the 60 % point is reached (Note 5). After the rapid-rise period (about 4 to 5 min), record temperatures at 1-min intervals on the minute until the difference between successive readings has been constant for 5 min.

NOTE 6—Use a magnifier and estimate all readings (except those during the rapid rise period) to the nearest 0.002°C (0.005°F) when using ASTM Bomb Calorimeter Thermometer 56C (56F). Estimate Beckmann thermometer readings to the nearest 0.001°C. Tap mercurial thermometers with a pencil just before reading to avoid errors caused by mercury sticking to the walls of the capillary.

NOTE 7—When the approximate expected rise is unknown, the time at which the temperature reaches 60 % of the total can be determined by recording temperatures at 45, 60, 75, 90, and 105 s after firing and interpolating.

11.7 *Observations, Adiabatic Jacket Method*—Assemble the calorimeter in the jacket and start the stirrer. Adjust the jacket temperature to be equal to or slightly lower than the calorimeter, and run for 5 min to obtain equilibrium. Adjust the jacket temperature to match the calorimeter with ±0.01°C (0.02°F) and hold for 3 min. Record the initial temperature (Note 6) and fire the charge. Adjust the jacket temperature to match that of the calorimeter during the period of rise, keeping the two temperatures as nearly equal as possible during the rapid rise; and adjusting to within ±0.01°C (0.02°F) when approaching the final equilibrium temperature. Take calorimeter readings at 1-min intervals until the same temperature is observed in three successive readings. Record this as the final temperature. Do not record time intervals since they are not critical in the adiabatic method.

11.8 *Analysis of Bomb Contents*—Remove the bomb and release the pressure at a uniform rate, in such a way that the operation will require not less than 1 min. Examine the bomb interior and discard the test if unburned sample or sooty deposits are found. Carefully wash the interior of the bomb including the capsule with distilled or deionized water containing the titration indicator until the washings are free of acid. Collect the washings in a beaker and titrate the washings with standard carbonate solution. Remove and measure or weigh the combined pieces of unburned firing wire, and subtract from the original length or weight to determine the wire consumed in firing. Determine the sulfur content of the sample by any of the procedures described in Test Methods E 775.

12. Calculation

12.1 *Temperature Rise in Isothermal Jacket Calorimeter*—Using data obtained as prescribed in 11.6, compute the temperature rise, T, in an isothermal jacket calorimeter as follows:

$$T = T_c - T_a - r_1(b - a) - r_2(c - b)$$

where:

T = corrected temperature rise,
a = time of firing,
b = time (to nearest 0.1 min) when the temperature rise reaches 60 % of total,
c = time at beginning of period in which the rate of temperature change with time has become constant (after combustion),
T_a = temperature at time of firing, corrected for thermometer error (Note 7),
T_c = temperature at time c, corrected for thermometer error (Note 7),
r_1 = rate (temperature units per minute) at which temperature was rising during 5-min period before firing, and
r_2 = rate (temperature units per minute) at which temperature was rising during the 5-min period after time c. If the temperature is falling, r_2 is negative and the quantity $r_2 (c - b)$ is positive.

12.2 *Temperature Rise in Adiabatic Jacket Calorimeter*—Using data obtained as prescribed in 11.7 compute the corrected temperature rise, T, as follows:

$$T = T_f - T_a$$

where:

T = corrected temperature rise, °C or °F,
T_a = initial temperature when charge was fired, corrected for thermometer error (Note 8), and
T_f = final temperature corrected for thermometer error.

NOTE 8—With all mercury-in-glass thermometers, it is necessary to make the following corrections if the total heat value is altered by 12 J/g or more. This represents a change of 0.001°C (0.002°F) in a calorimeter using approximately 2000 g of water. The corrections include the calibration correction as stated on the calibration certificate, the setting correction for Beckman thermometers according to the directions furnished by the calibration authority, and the correction for emergent stem. Directions for these corrections are given in Appendix X2.

12.3 *Thermochemical Corrections* (Appendix X3)—Compute the following for each test:

e_1 = correction for the heat of formation of HNO_3, J. Each millilitre of standard alkali is equivalent to 20.0 J.
e_2 = correction for heat of formation of H_2SO_4, J
= 55.2 × percent of sulfur in sample × weight of sample, g.
e_3 = correction for heat of combustion of firing wire, J (Note 10)
= 9.6 J/cm or 5980 J/g for No. 34 B & S gage Chromel C
= 11.3 J/cm or 7330 J/g for No. 34 B & S iron wire.
e_4 = correction for ignition energy of platinum or palladium if measured and corrected for.

NOTE 9—There is no correction for platinum or palladium wire, provided the ignition energy is constant.

12.4 *Calorific Value:*

12.4.1 Calculate the gross calorific value (gross heat of combustion) as follows:

$$H_s = [(T)(E) - e_1 - e_2 - e_3 - e_4]/g$$

where:

H_s = gross calorific value, J/g,
T = corrected temperature rise as calculated in 12.1 or 12.2, °C or °F, consistent with the water equivalent value,
E = energy equivalent (see Section 10),
e_1, e_2, e_3, e_4 = corrections as prescribed in 12.3, and
g = weight of sample, g.

12.4.2 Calculate the net calorific value (net heat of combustion) as follows:

$$H_i = H_s - 23.96\ (H \times 9)$$

where:

H_i = net calorific value (net heat of combustion), J/g,
H_s = gross calorific value (gross heat of combustion), J/g, and
H = total hydrogen, %.

13. Precision and Bias[10]

13.1 *Precision*—The standard deviations of individual determinations, in Btu/lb, are:

Average	Within-laboratory	Between-laboratories
HHV-1:		
6400	27.1	135.5
5200	48.8	239.6
HHV-2:		
7900	32.3	118.0
7400	38.1	227.8
HHV-3:		
9700	111.3	290.4
9500	99.2	249.2
9300	40.3	67.6

13.2 These precision estimates are based on an interlaboratory study conducted in accordance with Practice E 180.

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[10] Supporting data are available on loan from ASTM Headquarters. Request RR:E38-1000.

APPENDIXES

(Nonmandatory Information)

X1. CALCULATION OF STANDARD DEVIATIONS FOR CALORIMETER STANDARDIZATION

X1.1 The example given in Table X1.1 illustrates the method of calculating standard deviations for calorimeter standardizations.

TABLE X1.1 Standard Deviations for Calorimeter Standardization[A]

Standardization Number	Column A Water Equivalent, (Btu/lb) × (g/°C)	Column B Code to 4400 (Column A-4400)	Column C (Column B)^2
1	4412	12	144
2	4407	7	49
3	4415	15	225
4	4408	8	64
5	4404	4	16
6	4406	6	36
7	4409	9	81
8	4410	10	100
9	4412	12	144
10	4409	9	81
Sum		92	940

Average = $\bar{x}^2$ = x/10 = (92/10) + 4400 = 4409
Variance = s^2 = Column C − (Column B)$^2/n/n - 1$ = 940 − $(92)^2$/10/9 = 10.4
Standard deviation, s = Variance = 10.4 = 3.22

[A] In this example the values of water equivalent are typical for a calorimeter calibrated such that the water equivalent multiplied by the temperature rise in °C/g of sample will give the calorific value of the sample in Btu/lb.

X2. THERMOMETER CORRECTIONS

X2.1 It is necessary to make the following corrections in the event they result in an equivalent change of 0.001°C or more.

X2.1.1 *Calibration Correction* shall be made in accordance with the calibration certificate furnished by the calibration authority.

X2.1.2 *Setting Correction* is necessary for the Beckmann thermometer. It shall be made in accordance with the directions furnished by the calibration authority.

X2.1.3 *Differential Emergent Stem Correction*—The calculation depends upon the way the thermometer was calibrated and how it is used. The following two conditions are possible:

(*a*) *Thermometers Calibrated in Total Immersion and Used in Partial Immersion*—This emergent stem correction is made as follows:

$$\text{Correction} = K(t_c - t_a)\,(t_c + t_a - L - T)$$

where:

K = 0.00016 for thermometers calibrated in °C,
0.00009 for thermometers calibrated in °F,
L = scale reading to which the thermometer was immersed,
T = mean temperature of emergent stem,
t_a = initial temperature reading, and
t_c = final temperature reading.

NOTE X2.1: *Example*—Suppose the point L, to which the thermometer was immersed was 16°C; its initial reading, t_a, was 24.127°C, its final reading, t_c, was 27.876°C, the mean temperature of the emergent stem, T, was 26°C,

then:
Differential stem correction

$$= +0.00016\ (28 - 24)\ (28 + 24 - 16 - 26)$$
$$= +\ 0.006°\text{C}$$

(*b*) *Thermometers Calibrated and Used in Partial Immersion but at a Different Temperature than the Calibration Temperature*—This emergent stem correction is made as follows:

$$\text{Correction} = K\,(t_c - t_a)\,(t_1 - t^\circ)$$

where:

K = 0.00016 for thermometers calibrated in °C,
0.00009 for thermometers calibrated in °F,
t_a = initial temperature reading,
t_c = final temperature reading,
t_1 = observed stem temperature, and
t° = stem temperature at which the thermometer was calibrated.

NOTE X2.2: *Example*—Suppose the initial reading, t_a, was 80°F, the final reading, t_c, was 86°F, and that the observed stem temperature, t_1, was 82°F, and the calibration temperature, t°, was 72°F; then:
Differential stem correction

$$= 0.00009\ (86 - 90)(82 - 72)$$
$$= 0.005°\text{F}$$

X3. THERMOCHEMICAL CORRECTIONS

X3.1 *Heat of Formation of Nitric Acid*—A correction (e^1, in 12.3) of 20 J is applied for each 1 mL of standard Na_2CO_3 solution used in the acid titration. The standard solution (0.34 *N*) contains 18.02 g of Na_2CO_3/L. This correction is based on assumption that all the acid titrated is HNO_3 formed by the following reaction: $\frac{1}{2}\ N_2$ (g + $\frac{5}{4}\ O_2$ (g) + $\frac{1}{2}\ H_2O$ (l) = HNO_3 (in 500 mol H_2O), and (*2*) the energy of formation of 1 mol of HNO_3 is approximately 500 mol of water under bomb conditions is 14.1 kcal/mol.[6] When H_2SO_4 is also present part of the correction for H_2SO_4 is contained in the e_1 correction and the remainder in the e_2 correction.

X3.2 *Heat of Formation of Sulfuric Acid*—By definition the gross calorific value is obtained when the product of the combustion of sulfur in the sample is SO_2 (g). However, in actual bomb combustion processes, the sulfur is found as H_2SO_4 in the bomb washings. A correction (e_2 in 12.4.1) of 55.2 J is applied for each percent of sulfur in the 1-g sample, that is converted to H_2SO_4. This correction is based upon the energy of formation of H_2SO_4 in solutions such as will be present in the bomb at the end of a combustion. This energy is taken as −70.5 kcal/mol.[11] A correction, of 2 × 14.1 kcal/mol of sulfur was applied in the e_1 correction, so the additional correction necessary is 70.5 − (2 × 14.1) = 42.3 kcal/mol or 5520 J of sulfur in the sample (55.2 J × weight of sample in grams × % sulfur in sample).

X3.2.1 The value of 5520 J/g of sulfur is based on a coal containing about 5 % sulfur and about 5 % hydrogen. The assumption is also made that the H_2SO_4 is dissolved entirely in water condensed during combustion of the sample.[12] If a 1-g sample of such a fuel is burned, the resulting H_2SO_4 condensed with water formed on the walls of the bomb will have a ratio of about 15 mol of water to 1 mol of H_2SO_4. For this concentration the energy of the reaction.

$$SO_2\ (g) + \tfrac{1}{2}O_2\ (g) + H_2O\ (l) = H_2SO_4\ (\text{in } 15 \text{ mol } H_2O)$$

under the conditions of the bomb process is −70.5 kcal/mol.

X3.2.2 Basing the calculation upon a sample of comparatively large sulfur content reduces the overall possible errors, because for smaller percentages of sulfur the correction is smaller.

X3.3 *Fuse Wire*—Calculate the heat in SI units contributed by burning the fuse wire in accordance with the directions furnished by the supplier of the wire. For example, the heat of combustion of No. 34 B & S gage Chromel C wire

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[11] Calculated from data in National Bureau of Standards Circular 500.

[12] Mott, R. A., and Parker, C., "Studies in Bomb Calorimetry IX-Formation of Sulfuric Acid," *Fuel*, Vol 37, 1958, p. 371.

is equivalent to 9.6 J/cm or 5980 J/g and that of No. 34 B & S gage iron wire is equivalent to 11.3 J/cm or 7330 J/g. There is no correction for platinum or palladium wire provided the ignition energy is constant.

X4. REPORTING RESULTS IN OTHER UNITS

X4.1 *Reporting Results in British Thermal Units (Btu) per Pound*—The gross calorific value can be expressed in British thermal units by using the thermochemical correction factors in Table X4.1 and the water equivalent expressed in (Btu/lb) × (g/°C).

TABLE X4.1 Thermochemical Correction Factors (Units in BTU)

Correction	Multiplication Factor	Multiply by
e_1 (HNO_3)	10.0	mL of 0.394 N Na_2CO_3 solution
e_2 (H_2SO_4)	23.7	% of sulfur in sample times weight of sample in grams
e_3 (fuse wire)	4.1 or	cm of No. 34 B & S gage Chromel C wire
	2570	weight (g) of Chromel C wire
e_3 (fuse wire)	4.9 or	cm of No. 34 B & S gage iron wire
	3150	weight (g) of iron wire

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Standard Methods of
CONDUCTING STRENGTH TESTS OF PANELS FOR BUILDING CONSTRUCTION[1]

This standard is issued under the fixed designation E 72; 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.

INTRODUCTION

Sound engineering design of structures, using existing or new materials requires accurate technical data on the strength and rigidity of the basic elements employed in various construction systems. It is the purpose of these test methods to provide a systematic basis for obtaining engineering data on various construction elements and structural details of value to designers, builders, building officials, and others interested in this field. The results should closely approximate the performance in actual service.

1. Scope

1.1 These methods cover the following procedures for determining the structural properties of segments of wall, floor, and roof constructions:

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1.2 Metric units are to be considered as the primary standard units.

2. Applicable Documents

2.1 *ASTM Standards:*

E 4 Practices for Load Verification of Testing Machines[2]

E 73 Methods of Testing Truss Assemblies[3]

E 564 Method of Static Load Test for Shear Resistance of Framed Walls for Buildings[3]

E 575 Practice for Reporting Data from Structural Tests of Building Constructions, Elements, Connections, and Assemblies[3]

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[1] These methods are under the jurisdiction of ASTM Committee E-6 on Performance of Building Constructions and are the direct responsibility of Subcommittee E 06.12 on Structural Performance of Vertical Structures.

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[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 04.07.

E 661 Test Method for Performance of Wood and Wood-Based Floor and Roof Sheathing Under Concentrated Static and Impact Loads[3]

E 695 Method for Measuring Relative Resistance of Wall, Floor, and Roof Constructions to Impact Loading[3]

3. Test Specimens

3.1 *Size*—The specimens shall be representative as to material and workmanship and shall be as large as practicable to minimize the effect of variations in the material and workmanship, in order to obtain results representative of the construction. Obviously, the size of the specimens shall be limited to the size that can be tested in the larger testing machines available in a well equipped laboratory, and which can be subjected to loads in accordance with good testing procedure, and for which the deformation can be measured with sufficient accuracy.

3.2 *Length or Height*—The length or height of specimen for each element shall be chosen to conform to the length or height of that element in actual use.

3.3 *Width*—The width of specimen shall be chosen, insofar as possible, to include several of the principal load-carrying members to ensure that the behavior under load will simulate that under service conditions. With the exception of specimens for the racking load test, the nominal width of wall specimens shall be 1.2 m (4 ft). The actual width of specimens shall be a whole number multiplied by the spacing of the principal load-carrying members except for prefabricated panels, for which the actual width shall be the width of panel used. If the structural properties of a particular construction are to be compared with another construction, there should not be a great difference in the actual widths of the specimens.

3.4 *Age*—Constructions, such as concrete and masonry (brick, structural clay tile, concrete block) for which the structural properties depend upon the age of the specimen, shall be tested not less than 25 days nor more than 31 days after fabrication. This age requirement applies also to plastered and stuccoed constructions.

4. Loading

4.1 *Apparatus*—The testing machine or load-measuring apparatus shall comply with the requirements prescribed in Methods E 4.

4.2 *Application of Load*—Apply the load to all of the specimens in any test in increments so chosen that a sufficient number of readings will be obtained to determine definitely the load-deformation curve (see Section 6). Record the initial reading of the load and the reading of the deformation, either with no load on the specimen or under a small initial load. Increase the load to the first increment and record the deformation. Unless otherwise specified, decrease the load to the initial load and record the set (sometimes designated "permanent set"). Increase the load to two increments and record the set, when it is released to the initial load. Follow this sequence of readings for three increments, four increments, etc., of load. When for each specimen the behavior of the specimen under load indicates that the specimen might fail suddenly and damage the deformation-measuring apparatus, remove this apparatus from the specimen and increase the load continuously until the maximum load that can be applied to the specimen is determined.

4.3 *Duration of Load Application*—Except for racking tests, after each increment of load is applied, maintain the load level as constant as possible for a period of 5 min (see Note 1). Take deformation readings as soon as practical after load application, at the end of the 5-min period under constant load, and immediately and at the end of the 5-min period after any partial or complete load release. Plot initial and 5-min readings in the form of load-deformation curves. Maintain complete load-deformation-time records throughout the test. If application of a given load is required for a certain period, such as 24 h, take deformation readings at the beginning, at intervals during this period, and at the end of this period, to allow the satisfactory plotting of a time-deformation curve for the complete period.

NOTE 1—Reasons for the 5-min application of constant-level increment loads are as follows:

(1) To permit the assembly to come to a substantial rest prior to taking the second set of readings (Depending on the method employed for applying the test load, it may be necessary to continue, at a reduced rate, the motion of the loading device in order to maintain the constant load level during the 5-min period.)

(2) To provide sufficient time for making all observations. (Longer time intervals may be required under certain conditions.)

(3) To observe any time-dependent deformation

or load redistribution, or both, and to record accurately the load level when time-dependent deformation starts, that is, at the divergence of the immediate and delayed load-deformation curves. This load level may, under certain conditions, have an important bearing on the design load.

(4) To be able to stop the test, if this should be desirable, prior to total failure, after initial failure has been anticipated as a result of the observations.

(5) To assure uniformity in test performance and consistency in test results.

5. Deformation Measurements

5.1 Measure the deformations with sufficient precision to define the load-deformation relationship, and report at least to the nearest 0.25 mm (0.01 in.). The deformation-measuring apparatus specified for any loading may be replaced by other apparatus, provided that it permits readings of deformation that are equivalent in accuracy to those from the specified apparatus.

6. Reports

6.1 Show the results of each of the tests graphically, as illustrated in Fig. 1. Plot loads as ordinates and the deformations as abscissas for all tests. There shall be at least three specimens for each test, and the results for each test shall be shown on the same graph. Show the points for deformation under load by open circles and those for set by solid circles. Average the three values for either the deformation or the set and plot this average value in pencil on the graph. Draw a smooth curve among the average points to show the average behavior of the construction. The load-deformation curves shall be continuous lines and the load-set curves shall be dashed lines. Although the particular specimen for each point on the graph is not designated, record it on the laboratory data sheets. If readings are obtained under greater loads for some specimens than for others, plot all the values, but draw the curves only to the average values for which there are three values.

6.2 Prepare the test report in accordance with Recommended Practice E 575.

7. Precision and Accuracy

7.1 No statement is made either on the precision or on the accuracy of these methods due to the variety of materials and combinations of materials involved.

TESTING WALLS

8. Significance

8.1 The procedures described are those that will test the behavior of segments of wall construction under conditions representative of those encountered in service. Performance criteria based on data from those procedures can ensure structural adequacy and service life.

9. Compressive Load

9.1 *Test Specimens*—Tests shall be made on three like specimens, each having a height equal to the length of the element and a nominal width of 1.2 m (4 ft) (see Section 3).

9.2 *Apparatus*—The apparatus shall be assembled as shown in Fig. 2 and shall conform to the detailed requirements for component parts prescribed in 9.2.1 and 9.2.2, or the equivalent.

9.2.1 *Compressometer*—A bracket shall be attached to the specimen near the upper end, supporting a metal rod. A bracket shall also be attached to the specimen near its lower end, supporting a dial micrometer with the spindle up and the gage length shall be recorded. The conical end of the rod shall seat in a hole in the end of the spindle and the rod and spindle shall be held in contact by stretched rubber bands. The dial shall be graduated to 0.025 mm (0.001 in.).

9.2.2 *Deflectometer*—A fine wire shall be attached to a clamp near the upper end of the specimen. The free end connected to stretched rubber bands shall be attached to a clamp near the lower end of the specimen. A mirror having a paper scale one-half the width of the mirror shall be attached horizontally to the edge of the specimen at midheight. The scale shall be graduated to 2.5 mm (0.1 in.).

9.3 *Procedure:*

9.3.1 *Loading*—Test the specimen as a column having a flat end at the bottom (Fig. 2). Apply compressive loads to a steel plate covering the upper end of the specimen. Apply the load uniformly along a line parallel to the inside face, and one-third the thickness of the specimen from the inside face. For wood construction, a rate of loading corresponding to a movement of the testing machine crosshead of nominally 0.8 mm/min (0.03 in./min) has been found satisfactory.

9.3.2 *Load-Deformation Data*—Attach four compressometers to the faces of the specimen, one near each corner of the specimen as shown in Fig. 2, to measure the shortening of the specimen. Record the readings to the nearest 0.025 mm (0.001 in.).

9.3.3 *Lateral Deflection*—Attach two deflectometers, one to each edge of the specimen, as shown in Fig. 2. Record the readings, when the image of the wire coincides with the wire, to the nearest 0.25 mm (0.01 in.).

9.4 *Calculations and Report:*

9.4.1 *Deformation*—For each compressometer, calculate the shortening under each load as the difference between the reading of the compressometer when the load is applied and the initial reading. Calculate the shortening of the specimen as the average of the shortenings for each of the four compressometers multiplied by the ratio: specimen length divided by the compressometer gage length. Obtain the sets in a similar manner.

9.4.2 *Lateral Deflection*—Calculate the lateral deflection and the lateral set under each load for each deflectometer as the difference between the reading of the deflectometer when the load is applied and the initial reading. Calculate the lateral deflection and lateral set for the specimen as the average of the lateral deflection and lateral set of the two deflectometers.

9.4.3 *Data Presentation*—Record the maximum load for each specimen and report the results of load-deformation and load-deflection measurements in the form of a graph in accordance with Section 6. Report gage lengths of all deflection or deformation gages.

10. Tensile Load

10.1 *Test Specimens*—Tests shall be made on three like specimens, each having a height equal to the length of the element and a nominal width of 1.2 m (4 ft) (see Section 3).

10.2 *Apparatus*—The apparatus preferably shall be assembled in a vertical testing machine and shall conform to the detailed requirements for component parts prescribed in 9.2.1 and 9.2.2, or the equivalent, with the exception that the compressometers prescribed in 9.2.1 shall be replaced by extensometers which shall be like the compressometers but so adjusted before load is applied that the stretch of the specimen can be measured.

10.3 *Procedure:*

10.3.1 *Loading*—Test the specimen as a tension specimen by uniform application of tensile forces along the line of the fastenings at the top and the bottom of the wall in a building. The top and bottom pulling fixtures may be attached to the specimen by fastenings similar to those used in a building, provided that, under the maximum load, failure of the specimen occurs between the top and the bottom of the specimen, not in either the pulling fixtures or the fastenings. If, under the tensile load, failure occurs either in a pulling fixture or in a fastening, the results of the test determine only the properties of the fixtures or the fastenings, not of the wall construction. When the failure occurs in fastenings, the tensile load indicates the maximum tensile strength of the construction that can be realized in actual service unless improved fastenings are provided.

10.3.1.1 *Masonry Constructions*—The construction may be continued upward beyond the top of the specimen and downward below the bottom of the specimen to enclose attachments for the pulling fixtures.

10.3.1.2 *Framed Wall Constructions*—If the construction has studs (either of wood or metal) the studs may be extended upward and downward beyond the top and bottom of the specimen and attached to the pulling fixtures. If the framed wall has plates at the top and the bottom, attach the pulling fixtures to the plates in the specimen.

10.3.2 *Load-Deformation Data*—Attach four extensometers to the faces of the specimen, one near each corner, as shown in Fig. 2, to measure the stretch of the specimen. Record the readings to the nearest 0.025 mm (0.001 in.).

10.3.3 *Lateral Deflection*—Attach two deflectometers, one to each edge of the specimen, as shown in Fig. 2. Record the readings, when the image of the wire coincides with the wire, to the nearest 0.25 mm (0.01 in.). Lateral deflection (if any) may be caused by nonaxial loading of the specimen.

10.4 *Calculations and Report*—For tensile loads, the calculations and report shall be similar to those required for compressive loads (see 9.4).

11. Transverse Load—Specimen Horizontal

11.1 *Test Specimens*—Tests shall be made on three like specimens on symmetrical assemblies and six like specimens on unsymmetrical assemblies, each having a length equal to the length of the element and a nominal width of 1.2 m (4 ft) (see Section 3).

11.2 *Apparatus*—The apparatus shall be assembled as shown in Fig. 3 and shall conform to the detailed requirements for component parts prescribed in 11.2.1 through 11.2.3, or the equivalent.

11.2.1 *Supports*—Two steel rollers with a steel plate between each supporting roller and the specimen.

11.2.2 *Loading Assembly*—Two steel rollers with a steel plate between each loading roller and the specimen.

11.2.3 *Deflection Gage*—A frame shall be placed on the upper face of the specimen. To prevent stresses deforming the frame as the specimen deforms under load, this frame shall rest on three hardened steel balls each supported by a steel block on the face of the specimen. Two of the balls shall be placed in a line vertically above one support and the third ball vertically above the other support. Two dial micrometers, one near each longitudinal edge of the specimen, shall be attached to the frame at midspan. The spindles shall rest on the upper face of the specimen. The micrometers shall be graduated to 0.025 mm (0.001 in.).

11.3 *Procedure:*

11.3.1 *Loading*—Use "two-point" loading for transverse load tests. Test the specimen as a simple beam (Fig. 3) on a span 150 mm (approximately 6 in.) less than the specimen length. Apply two equal loads, each at a distance of one quarter of the span from the supports, toward the middle of the span. For wall specimens tested horizontally (Fig. 3), the load on the specimen shall include the weight of specimen between the supports. Apply the transverse loads to the outside face for three of the specimens and to the inside face for three of the specimens. For symmetrical assemblies, test only three specimens.

11.3.1.1 Uniformly distributed loading may be used instead of quarter-point loading, if a satisfactory method is available. The transverse strength for any span may be greater for some constructions under uniformly distributed load than under loads applied at the quarter-points of the span. Transverse load, uniformly distributed, may be applied by air pressure, either in a bag or in a chamber having the specimen as one face. Support specimens tested under uniform loading by rollers as for quarter-point loading.

11.3.1.2 The bag method of loading is shown schematically in Fig. 4. Connect a reaction platform parallel to the face to be loaded and wider than the specimen to the supports by tie rods. Place an airtight bag of rubberized cloth as wide as the specimen and as long as the span between the specimen and the reaction platform. Apply transverse load to the specimen by increasing the air pressure in the bag. Measure the pressure by means of a manometer. Water is usually the liquid in the manometer, but the specific gravity of the liquid shall be such that the error in pressure readings does not exceed 1 %.

11.3.1.3 When the chamber method of loading is used with the specimen horizontal, place the specimen near the floor, which should be practically airtight. An airtight frame or curb shall surround the specimen closely and be about flush with the upper surface of the specimen. A rubber blanket covers the specimen, overlaps the frame, and is sealed so that it is reasonably airtight. Use a small vacuum pump or positive action exhaust blower to reduce air pressure between the specimen and floor. Measure the difference in pressure above and below the specimen by means of a manometer.

11.3.2 *Strength on Short Span*—The transverse strength of any construction increases as the span is shortened. If the strength of the construction for a shorter span is desired, do not compute it, but test the construction on the short span.

11.4 *Calculations and Report:*

11.4.1 *Load-Deflection Data*—For each micrometer, calculate the deflection under a given load as the difference between the reading to the nearest division of the micrometer when the load is applied and the initial reading. Calculate the deflection of the specimen for the span as the average of the deflections obtained from each of the two micrometers. Calculate the sets under the initial load by using a similar method. Record the maximum load for each specimen.

11.4.2 *Data Presentation*—Report the results in the form of a graph in accordance with Section 6.

12. Transverse Load—Specimen Vertical

12.1 *Test Specimens*—Tests shall be made on three like specimens on symmetrical assemblies and six like specimens on unsymmetrical assemblies each having a length equal to the length of the element and a nominal width of 1.2 m (4 ft) (see Section 3).

12.2 *Apparatus*—The apparatus shall be assembled as shown in Fig. 3 and shall conform to the requirements for component parts prescribed in 12.2.1 through 12.2.5, or the equivalent.

12.2.1 *Steel Channel.*

12.2.2 *Rollers*—Cylindrical rollers, two supporting rollers, two loading rollers.

12.2.3 *Screw Jack.*

12.2.4 *Ring Dynamometer.*

12.2.5 *Deflectometers*—Two taut-wire mirror-scale deflectometers similar to those described in 9.2.2.

12.3 *Procedure*—Transverse loads cannot be applied satisfactorily to some wall constructions, such as masonry, with the specimen in a horizontal position. For such constructions, apply the loads with the specimen in a vertical position, as shown in Fig. 3, thus simulating service conditions. The specimen, on a steel channel, shall rest on cylindrical rollers to prevent restrained end conditions. The axes of the rollers shall be parallel to the faces of the specimen. The two supporting rollers shall be in contact with the vertical surface of the frame and each roller shall rest horizontally on sponge rubber about 10 mm (0.4 in.) thick to prevent longitudinal restraint. Each of the two loading rollers shall also rest on sponge rubber. Apply the loads horizontally by a screw jack and measure by a ring dynamometer between the jack and the specimen. The error in the load indicated by the dynamometer shall not exceed 1 %. Attach two taut-wire mirror-scale deflectometers to the specimen, one to each vertical edge.

12.3.1 Apply the transverse load to the outside face for three of the specimens, and to the inside face for three of the specimens. For symmetrical assemblies, test only three specimens.

12.3.2 When the Chamber Method of loading is used with the specimen vertical, the specimen forms one face of an airtight chamber from which the air is exhausted. If all four edges of the specimen bear on the chamber, this loading determines the strength of the specimen as a plate supported at the four edges, not the transverse strength as defined in these methods.

12.3.3 If a specimen tested by the chamber method, either horizontally or vertically, has an airtight cavity, vent each cavity to the low-pressure face by a hole in the face of the specimen not less than 5 mm (0.2 in.) in diameter, located where it will least affect the transverse strength of the specimen.

12.4 *Calculations and Report*—Calculate the results of test and report as described in 11.4, and report deflectometer readings to the nearest 0.25 mm (0.01 in.).

13. Concentrated Load

13.1 *Test Specimens*—Concentrated load tests shall be made on each transverse specimen after the transverse load tests, the concentrated load being applied to the same face to which the transverse load was applied.

13.2 *Apparatus*—The apparatus shall be assembled as shown in Fig. 5 and shall conform to the requirements for component parts prescribed in 13.2.1 through 13.2.3, or the equivalent.

13.2.1 *Steel Bar*—Steel bar having a diameter of 25.4 mm (1 in.) and the edge of the face contacting the specimen rounded to a radius of 1.3 mm (0.05 in.).

13.2.2 *Depth Gage*—The depth gage shall consist of a dial micrometer graduated to 0.025 mm (0.001 in.) mounted on a three-legged support. The support shall be notched to permit placing the micrometer directly adjacent to the bar and shall be long enough to permit placing the supporting legs on undisturbed areas of the face of the specimen.

13.2.3 *Loading Device*—Any convenient means for applying a compressive load up to 5 kN (1100 lbf) and means for measuring the load within 1 %.

13.3 *Procedure:*

13.3.1 *Loading*—Place the entire specimen or portion of the specimen on a horizontal support and properly level. Place the steel bar on the face of the specimen at what is judged to be the weakest place and, also, at what is judged to be the strongest place. Apply a load vertically downward to the upper surface of the bar. Continue loading until maximum load or 4.45 kN (1000 lbf) is attained.

13.3.2 *Depth of Indentation*—Measure the depth of indentation, by means of the depth gage, and record the reading of the micrometer to the nearest 0.025 mm (0.001 in.).

13.4 *Calculations and Report:*

13.4.1 *Depth of Indentation*—Calculate the depth of indentation (set) after a given load has been applied and the bar removed to the nearest 0.025 mm (0.001 in.) as the difference between the depth for that load and the initial reading of the micrometer before a load has been applied to the specimen.

13.4.2 *Data Presentation*—Report the results in the form of a graph in accordance with Section 6.

14. Racking Load—Evaluation of Sheathing Materials on a Standard Wood Frame

Note 2—If the test objective is to measure the performance of the complete wall, Method E 564 is recommended.

14.1 *Scope*—This test method measures the resistance of panels, having a standard wood frame, and sheathed with sheet materials such as structural insulating board, plywood, gypsum board, transite, etc., to a racking load such as would be imposed by winds blowing on a wall oriented at 90° to the panel. It is intended to provide a reliable, uniform procedure for determining the resistance to racking load provided by these sheet materials as commonly employed in building construction. Since a standard frame is employed, the relative performance of the sheathing is the test objective.

14.1.1 This test is conducted with standardized framing, loading procedures, and method of measuring deflection, as detailed in the method to ensure reproducibility. Provision is made for following the sheathing manufacturers' recommendations for attaching the sheathing to the frame, and for reporting the behavior of the specimen over its entire range of use.

14.1.2 In applying the results, due allowance shall be made for any variation in construction details or test conditions from those in actual service.

14.2 *Test Specimens:*

14.2.1 *Size and Number*—The test specimen shall be 2.4 by 2.4 m (8 by 8 ft) and the framing shall be constructed as shown in Fig. 6 and a minimum of three panels of each construction shall be tested. It is the intent of this test procedure to evaluate the stiffening effect of the sheathing material; therefore, the frame shall be constructed as nearly like the frames shown in Fig. 6 as possible. Frames shall be newly constructed for each test. All individual framing members shall be continuous. The moisture content of framing material shall be between 12 and 15 % when the panel is fabricated, and shall not vary by more than 3 % from the initial moisture content when the panel is tested.

14.2.2 *Application of Sheathing*—The method of applying the sheathing shall be exactly as specified by the manufacturer. The spacing of fasteners shall be as recommended. Fasteners shall be driven through the sheathing into only the outside stud of each corner post shown in Fig. 6. The importance of the attachment of sheathing to the framing cannot be overemphasized. Slight differences in edge clearances, angle of fastener, and amounts of penetration of heads of fasteners into the sheathing have appreciable effects on the results of test. Unless otherwise specified, fasteners shall be driven perpendicular to the surface of the sheathing with the center of each fastener the specified distance from the edge of the sheathing. Fasteners shall be driven so that the head of the fastener contacts the surface of the sheathing but not so deep as to crush the surface, unless specified differently by the manufacturers.

14.3 *Apparatus*—The apparatus shall be assembled as shown in Fig. 7. Load shall be measured by means of a testing machine, or a dynamometer attached to cables that load the specimen, or in linkage with a hydraulic jack used to apply load. The essential parts of the testing apparatus, exclusive of the loading frame, are as described in 14.3.1 through 14.3.5.

14.3.1 *Base and Loading Frame*—The test panel shall be attached to a timber or steel plate that is in turn attached rigidly to the base of the loading frame in such a manner that when the panel is racked, the sheathing will not bear on the loading frame. This member may be of any convenient cross section, but it shall be at least as long as the panel and not greater in width than the thickness of the frame, 89 mm (3½ in.). Means shall be provided to bolt or otherwise attach the sole plate of the panel firmly to this member. For illustrative purposes, two bolts are shown in Fig. 7. More may be used if required.

14.3.2 *Hold-Down*—A hold-down shall be provided as shown in Fig. 7 to overcome the tendency of one end of the panel to rise as the racking load is applied. Plates and rollers shall be provided between the test specimen and the hold-down so that the top of the specimen can deflect horizontally with respect to the bottom without unnecessary interference from the hold-down. Because the amount of tension in the rods of the hold-down may have an effect on the results of the test, nuts on the hold-down rods shall be tightened prior to load application so that the total force in each rod does not exceed 90 N (20 lbf) at the beginning of test as determined by previous calibration.

14.3.3 *Loading Apparatus*—Load shall be applied to the specimen through an 89 by 89-mm (3.5 by 3.5-in.) timber firmly bolted to the upper plates of the panel. Loading shall be a compressive force against the end of the timber attached to the upper plate. When a testing machine is used, pulleys and cables may be used to transmit the vertical movement of the tension head of the machine to the horizontal movement in the specimen.

14.3.4 *Lateral Guides*—Lateral guides shall be provided so that the specimen will deflect in a plane. The rollers should be bearing-supported to reduce friction to a minimum. The lateral guides shall be firmly attached to the loading frame. Plates for the rollers may be up to 300 mm (12 in.) in length as required.

14.3.5 *Indicating Dials*—Indicating dials, or scales and wires, shall be provided to measure the displacement of the different parts of the panel during test. The readings shall be recorded to the nearest 0.25 mm (0.01 in.). The locations of the dials shall be as shown in the lower left, lower right, and upper right corners of the side view of the test assembly in Fig. 7. The dial at the lower left, which is attached to the stud, measures any rotation of the panel, the dial at the lower right measures any slippage of the panel, and the dial at the upper right measures the total of the other two plus the deformation of the panel. Therefore, the horizontal deflection of the panel at any load is the reading of the dial at the upper right less the sum of the readings of the other two.

14.4 *Procedure:*

14.4.1 *Loading*—Apply the load continuously throughout test at a uniform rate of motion of the loading device used. The recommended speed of testing shall be such that the loading to 3.5 kN (790 lbf) total load shall be completed in not less than 2 min from the start of the test. The loading to 7.0 to 10.5 kN (1570 to 2360 lbf) total load and to failure shall employ the same rate of travel of the loading device as for the loading to 3.5 kN. Give the speed of testing used in the report of test.

14.4.2 *Loading Procedure*—Load the specimen in three stages to 3.5, 7.0, and 10.5 kN (790, 1570, and 2360 lbf) total load at a uniform rate.

14.4.2.1 To provide data to meet performance requirements, other values of total load may be included in the test procedure. Use the same rate of loading as for the loadings specified and indicate additional loadings evaluated and the results obtained in the report.

14.4.2.2 After the load of 3.5 kN (790 lbf) is placed on the specimen, remove all of the load and any residual deflection (set) in the panel noted. Then load the specimen to 7.0 kN (1570 lbf) and again remove the load and note any additional set; after this increase the loading to 10.5 kN (2360 lbf), remove the load again, and note the set. Apply load continuously for each of the increment loads specified above and obtain load-deflection data. Obtain these data for at least each 900 N (200 lbf) of loading. Obtain deflections during the loading cycle and, if desired, during the unloading cycle as well.

14.4.2.3 After the specimen is loaded as specified to 3.5, 7.0, and 10.5 kN (790, 1570, and 2360 lbf) load it again to failure or until the total deflection of the panel becomes 100 mm (4 in.). Obtain readings of deflection for the same intervals of load as were used for the other loadings.

14.5 *Calculations and Report:*

14.5.1 *Deformation*—For each dial, or other measuring device, calculate the movement under each racking load as the difference between the readings when load is applied and the initial readings at the start of the test. Calculate set readings as the difference between the readings when the load is removed and the initial readings.

14.5.2 *Data Presentation*—Report the deflections at 3.5, 7.0, and 10.5 kN (790, 1570, and 2360 lbf) and the set after loading to these amounts. Present load-deflection curves obtained during loading to failure and to 3.5, 7.0, and 10.5 kN in the form of a graph as prescribed in Section 6. Include maximum load and any observations on the behvaior of the panel during test and at failure. Express residual deflections (sets) as percentages of the deflections that produced the sets as well as in millimetres or inches. If the specimen fails, describe the visible failure. If the specimen has been subjected to any special conditioning prior to test, describe this treatment in detail. Describe in the report the sheathing used, the method of applying the sheathing, the type and spacing of fasteners, and the method and rate of loading employed.

15. Racking Load—Evaluation of Sheathing Materials (Wet) on a Standard Wood Frame

15.1 *Scope*—This test has been developed to simulate the degree of wetting possible during construction of a structure when, because of rain, the framing and sheathing may be wetted on one or both sides. Both sides of the wall panel are wetted because this represents the maximum exposure possible during the stage of construction before the structure is roofed.

15.2 *Test Specimens*—The test specimens shall conform in size and fabrication details to the requirements of 14.2.

15.3 *Specimen Conditioning*—Mount the fabricated test specimens or suspend them in a vertical position in such a manner as to prevent continuous immersion of the bottom edge of the specimen. Expose both sides of the test specimen to a water spray applied at or near the top along the entire length to ensure that the top of the specimen is being wetted. The spray shall have no jet action that cuts into the sheathing material, and the spray areas shall overlay sufficiently so that a continuous sheet of water flows down both surfaces of the specimen. Maintain the temperature of the water in the line to the spray nozzle at 24 ± 3°C (75 ± 5°F). Wet the specimens for a period of 6 h and then allow to dry for a period of 18 h. Dry in laboratory air, preferably at a temperature of 24 ± 3°C (75 ± 5°F). Make no attempt to increase the air movement over the specimens by fans or blowers. Subject the test specimens to two complete wetting and drying cycles and then a third wetting cycle.

15.3.1 No more than 2 h shall elapse between the completion of the third wetting cycle and the start of the racking test.

15.4 *Procedure*—Test the specimens in accordance with the procedure described in 14.4.

15.5 *Moisture Content Determination*—After the racking test is completed, cut moisture samples from the sheathing material, and determine moisture content on a weight basis with the moisture content expressed as a percentage of the oven dry weight in accordance with 15.5.1. Preferably, take five moisture content samples at least 100 by 150 mm (4 by 6 in.) in size from each 1.2 by 2.4-m (4 by 8-ft) sheathing panel of the test specimen: one from the center of each sheathing panel at the top and bottom edges, one from midlength on each side, and one from the panel center. Weigh the moisture content samples immediately upon being cut from the test specimen to an accuracy of not less than ±0.2 %. Carefully remove all loose particles from the sample before weighing. Then dry the samples to constant weight in an oven at 103 ± 2°C (217 ± 4°F). If large amounts of volatile matter or substances other than free water are removed from the sheathing material by drying at 103 ± 2°C, the sheathing material may be dried to constant weight at a lower temperature and the drying time and temperature given in the report.

15.5.1 *Calculation*—Calculate the moisture content as follows:

$$M = 100\,[(W - F)/F]$$

where:

M = moisture content, %,
W = initial weight, and
F = final weight when oven dry.

15.6 *Calculations and Report*—The report shall include the racking test data as specified in 14.5. It shall also include the line temperature of the water sprayed on the test specimens; the air temperature and relative humidity during the drying portion of the cycle; and the location of the moisture content samples and the moisture content of each.

TESTING FLOORS

16. Significance

16.1 The procedures outlined will serve to evaluate the performance of floor segments under conditions representative of those sustained in service. Performance criteria based on data from these procedures can ensure structural adequacy and effective service.

17. Transverse Load

17.1 *Test Specimens*—Tests shall be made on three like specimens, each having a length equal to the length of the floor panel and a nominal width of 1.2 m (4 ft) (see Section 3).

17.2 *Apparatus*—The apparatus shall conform to the requirements of 11.2.

17.3 *Procedure*—Conduct the test in accordance with 11.3 on transverse load tests of walls, except apply the loads only to the upper (finish floor) face of the specimen. If practicable, test floor specimens in the horizontal position. If tested in the vertical position, conduct the test in accordance with 12.1 through 12.4 on transverse load tests on walls in the vertical position. If tested in the vertical position, deduct transverse load equal to the weight of the specimen from each recorded load to obtain the applied load on the specimen.

17.3.1 *Strength on Short Span*—The transverse strength of any floor construction increases as the span is shortened. If the strength of the construction for a shorter span is desired, do not compute it, but test the construction on the shorter span.

17.4 *Calculations and Report*—Report the results as indicated in 11.4.

18. Concentrated Load

18.1 *Test Specimens*—Tests shall be made on each of the transverse specimens after the transverse tests are completed.

18.2 *Apparatus*—The apparatus shall conform to the requirements of 13.2.

18.3 *Procedure*—Conduct the test in accordance with 13.3 on concentrated load tests on walls, except apply the loads only to the upper (finish floor) face of the specimen.

18.4 *Calculations and Report*—Report the results as indicated in 13.4.

TESTING ROOFS

19. Significance

19.1 These procedures will serve to evaluate performance of roof segments under simulated service conditions. Roof trusses shall be evaluated under Methods E 73.

20. Transverse Load

20.1 *Test Specimens*—Tests shall be made on three like specimens, each having a length equal to the length of the roof panel and a nominal width of 1.2 m (4 ft) (see Section 3).

20.2 *Apparatus*—The apparatus shall conform to the requirements of 11.2.

20.3 *Procedure*—Conduct the test in accordance with 11.3 on transverse load tests of walls, except normally apply the loads only to the upper (weatherproofed) face of the specimen. The transverse strength of a roof construction under loads acting outward may appear to be less than the strength under loads acting inward. For such constructions, apply loads acting outward to specimens.

20.3.1 *Strength on Short Span*—The transverse strength of any roof construction increases as the span decreases. If the strength of the construction for a shorter span is desired, do not compute it, but test the construction on the shorter span.

20.4 *Calculations and Report*—Report the results as indicate in 11.4.

21. Concentrated Load

21.1 *Test Specimens*—Tests shall be made on each of the transverse specimens after the transverse tests are completed.

21.2 *Apparatus*—The apparatus shall conform to the requirements of 13.2.

21.3 *Procedure*—Conduct the test in accordance with 13.3 on concentrated load tests of walls, except apply the loads only to the upper (weatherproofed) face of the specimen.

21.4 *Calculations and Report*—Report the results as indicated in 13.4.

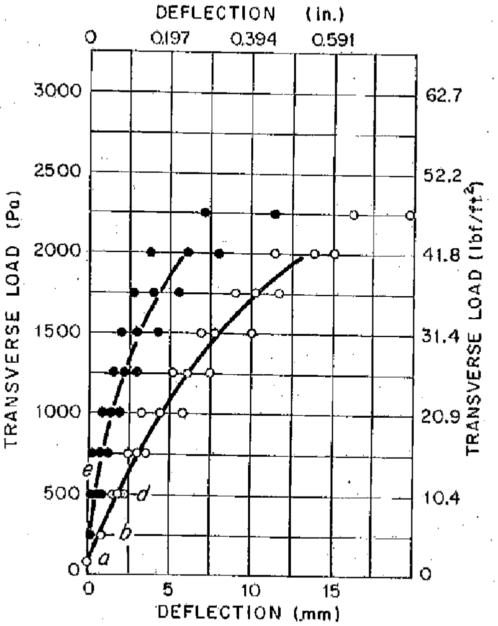


FIG. 1 Typical Graph Showing Results

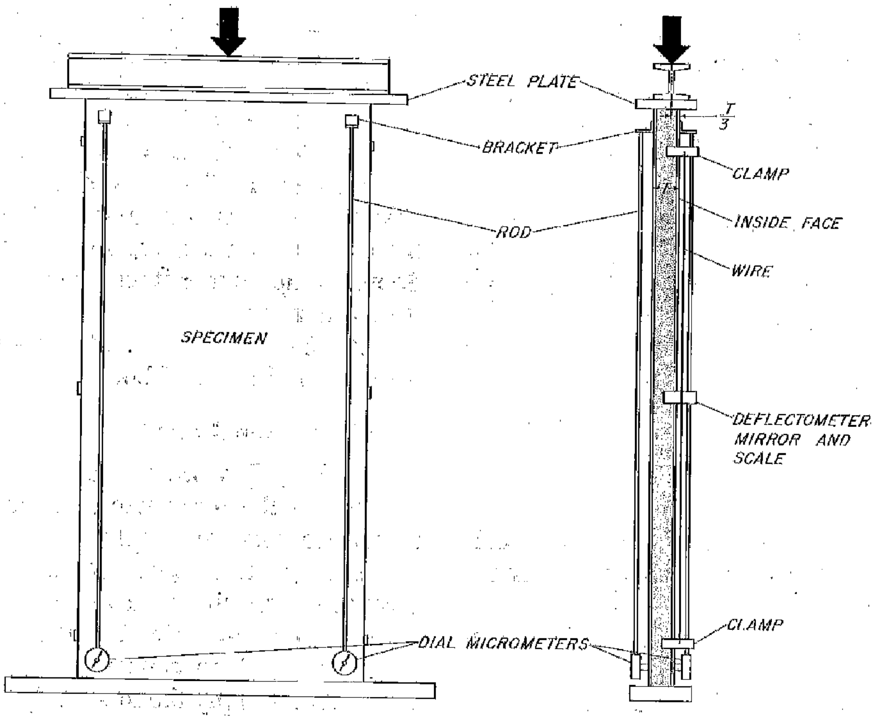
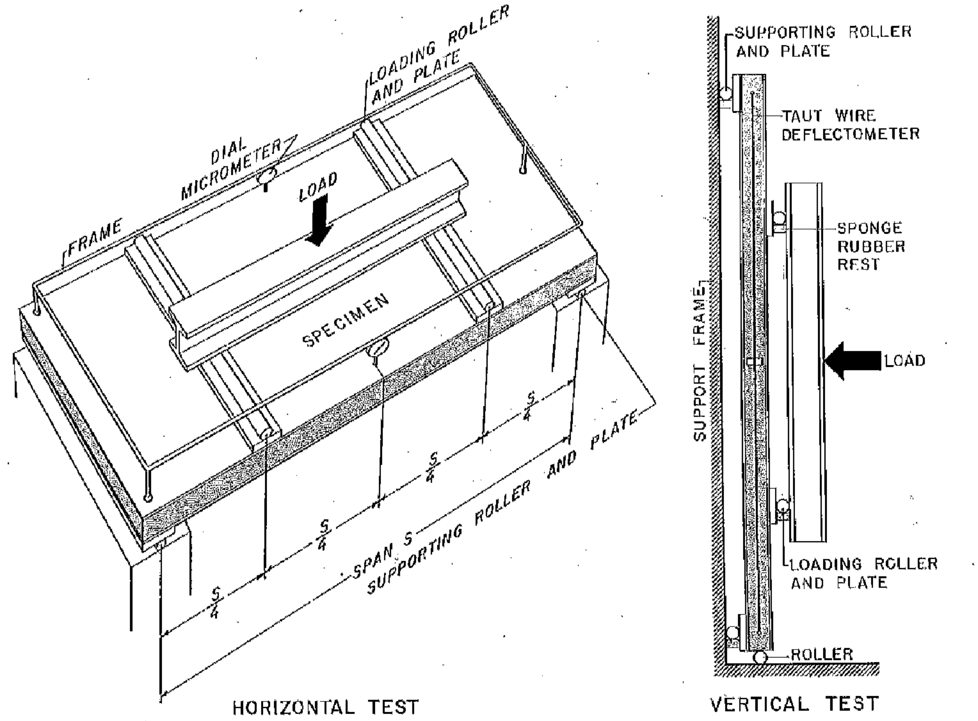


FIG. 2 Compressive Load Test on Wall Specimen



HORIZONTAL TEST

VERTICAL TEST

FIG. 3 Transverse Load Test on Wall Specimen

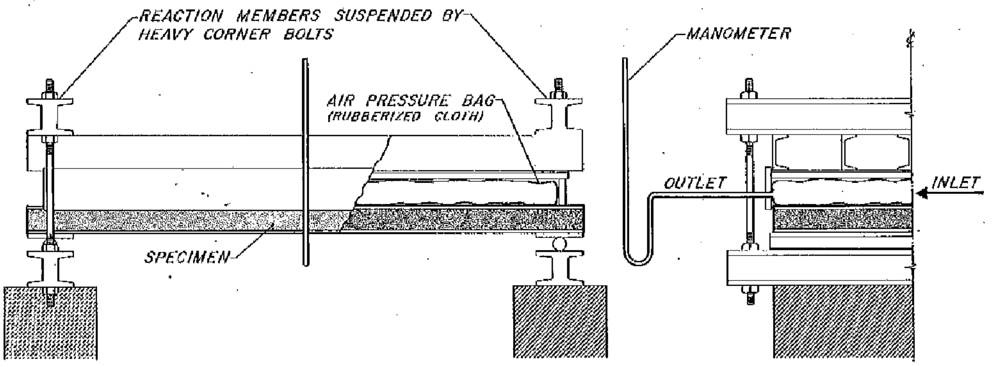


FIG. 4 Apparatus for Uniformly Distributed Transverse Load (Bag Method)

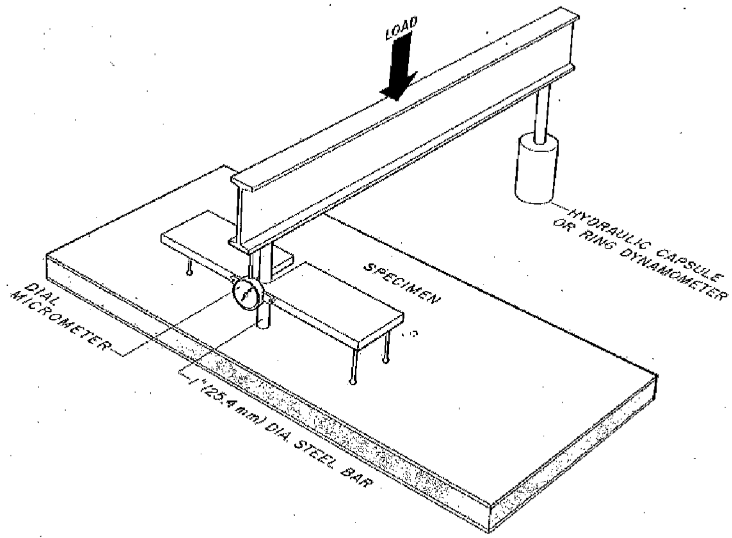
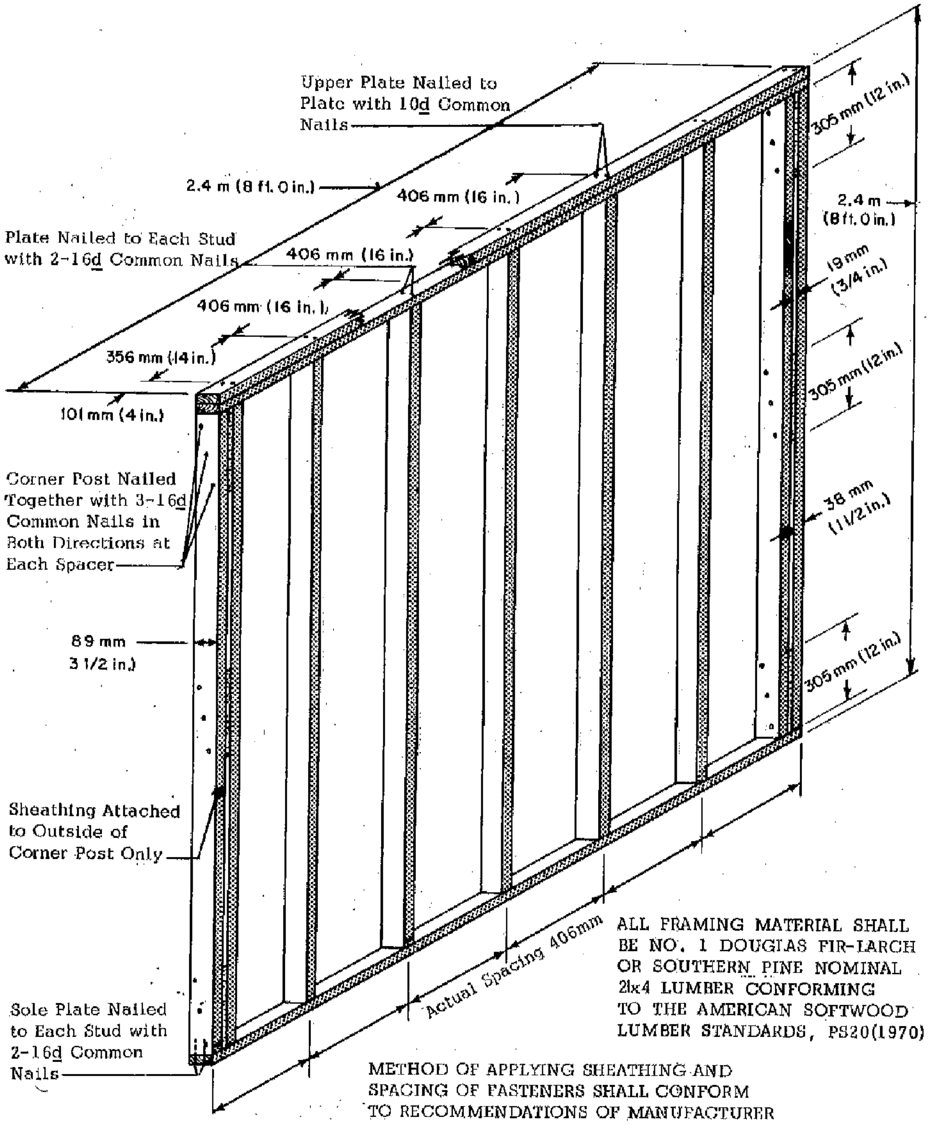


FIG. 5 Concentrated Load Test

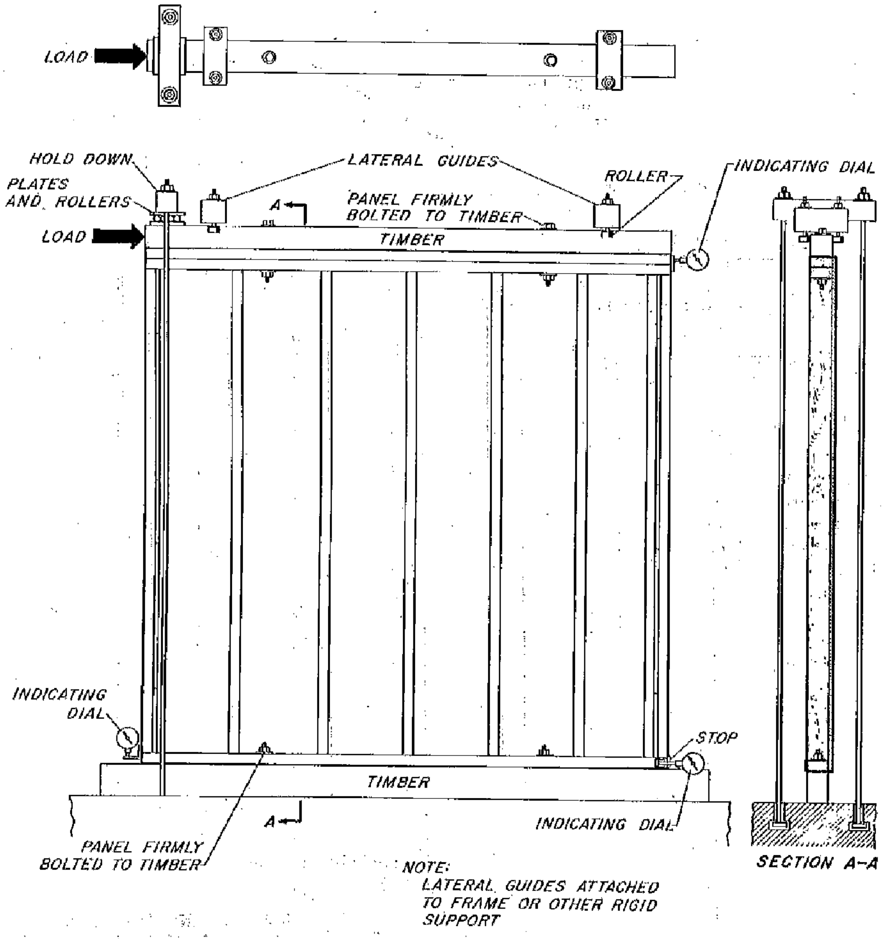


ALL FRAMING MATERIAL SHALL BE NO. 1 DOUGLAS FIR-LARCH OR SOUTHERN PINE NOMINAL 2x4 LUMBER CONFORMING TO THE AMERICAN SOFTWOOD LUMBER STANDARDS, PS20(1970)

METHOD OF APPLYING SHEATHING AND SPACING OF FASTENERS SHALL CONFORM TO RECOMMENDATIONS OF MANUFACTURER

NOTE—To eliminate test data that may be misleading, use lumber of average density for the species involved.

FIG. 6 Standard Wood Frame



NOTE: LATERAL GUIDES ATTACHED TO FRAME OR OTHER RIGID SUPPORT

FIG. 7 Racking Load Assembly

APPENDIX

(Nonmandatory Information)

X1. TECHNICAL INTERPRETATION

X1.1 It is the purpose of these test methods to provide a systematic basis for obtaining comparable engineering data on various construction elements and structural details of value to designers, builders, building officials, and others interested in this field.

X1.2 Subjecting complete structures to known loads is very expensive and requires much time; therefore, that method of carrying out investigations to establish structural properties is not likely to be used to any great extent. Such tests have the further disadvantage that only the strength of the weakest elements of a particular structure could be measured.

X1.3 For these reasons, it seems more practicable to apply loads to specimens that accurately reproduce a structural portion of a finished building. These portions of a building have been designated as “elements”; for example, floor, wall, roof, etc. For the procedure described in these methods, the elements have been restricted to those most important structurally. For each element, methods of loading are described that simulate the loads to which the element would be subjected under service conditions. It is believed that the results of these measurements on the structural elements will be more useful to architects and engineers than the results of tests on specimens of the materials from which the structure was fabricated, or the results of tests of the individual structural members. Although it may be impracticable to determine all of the structural properties of each element of a building, it is believed that the more important properties may be determined by tests described in these methods.

X1.4 The test method, involving the application of the loads in increments and the concurrent measurement of deformation and set, simulates, to some extent, the conditions of repeated loading under service conditions. Therefore, results by such a method of loading may be more useful than those obtained by increasing the load continuously throughout the test. The results from increment loading tests may show whether different portions of a construction act as a unit under load, whether the fastenings or bonds have adequate strength, or whether they rupture under repeated loads. For any engineering structure, including small houses, it is necessary not only that the strength be adequate, but also that the deformation under load shall not appreciably decrease the usefulness of the structure. If the working load and the allowable deformation for an element for a structure are known, constructions complying with these requirements may be selected by inspection of the graphs from tests of such constructions.

X1.5 A structure is elastic if, after a load has been applied and then removed, the set is inappreciable. If the set is small for an element of a building, it may be assumed that the construction has neither been damaged nor appreciably deformed by the load. The set, therefore, is another property that may be used when comparing different constructions and may be useful when selecting a construction for a particular purpose.

X1.6 The variations in the properties of a construction as used commercially for buildings, in all probability, will be greater than the variations for the three specimens tested as directed in these methods because these specimens will be all fabricated at the same time by the same workmen and from the same lot of material. This fact should be clearly indicated in any general report based on these test procedures.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

Document Name: ASTM E773: Standard Test Method for Seal Durability of Sealed Insulating Glass Units

CFR Section(s): 4 CFR 3280.403(d)(2)

Standards Body: American Society for Testing and Materials

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 773 – 97

An American National Standard

Standard Test Method for Accelerated Weathering of Sealed Insulating Glass Units[1]

This standard is issued under the fixed designation E 773; 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 test method covers procedures for testing the performance of preassembled permanently sealed insulating glass units against accelerated weathering.

1.2 This test method is applicable only to sealed insulating glass units, with one or two airspaces, fabricated for vision glass areas for use in buildings, such as sliding doors, windows, wall systems, and picture windows.

1.3 The unit construction used in this test method contains dimensions that are an essential component of the test. Deviations of glass or airspace sizes, or both, will affect the test results.

1.4 This test method is not applicable to sealed insulating glass units that are constructed from exterior vision materials other than glass.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *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.* A specific hazard statement is given in Note 2.

2. Referenced Documents

2.1 *ASTM Standards:*

C 1036 Specification for Flat Glass[2]

E 546 Test Method for Frost Point of Sealed Insulating Glass Units[3]

E 774 Specification for the Classification of the Durability of Sealed Insulating Glass Units[3]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *chemical dew point*—the temperature (Note 1) at which organic vapor or other chemical begins to condense on the interior glass surface of a sealed insulating glass unit.

Note 1—The temperature as indicated by the temperature indicator of frost point apparatus when constructed and used in accordance with test procedures described in Test Method E 546.

3.1.2 *frost point*—the temperature (Note 1) below freezing point of water (0°C (32°F)) at which visible frost begins to deposit on the interior glass surface of a sealed insulating glass unit.

4. Significance and Use

4.1 This test method is intended to provide a means for evaluating the durability of the sealing system of sealed insulating glass units. A round-robin test has been conducted, and additional data are being collected. The suitability of these test methods will be further evaluated as the analysis of test results is completed.

5. Apparatus

5.1 *For High Humidity Testing*:

5.1.1 *High-Humidity Test Chamber*—A chamber of convenient dimensions capable of maintaining 60 ± 3°C (140 ± 5°F) and 95 ± 5 % relative humidity.

5.2 *For Accelerated Weather Cycle Test*:

5.2.1 *Accelerated Weather Cycle Test Apparatus*[4]—The accelerated weather cycle test apparatus shall be essentially that shown in Figs. 1-3 to provide the cycle conditions and time frame indicated in Section 8.

5.2.2 *Ultraviolet Light Source*:

Note 2—**Warning:** Ultraviolet light sources used in this test method are harmful, especially to the eyes. Appropriate protective measures must be observed.

5.2.2.1 The source shall consist of two fluorescent black light lamps, Type F72T12BL/HO[5,6] (Note 3), for each test specimen located as shown in Fig. 3.

5.2.2.2 Each lamp must be replaced when its ultraviolet light intensity falls below 10 kW/m^2 when measured with a long-wave ultraviolet meter[5] in direct contact with the lamp.

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[1] This test method is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.22 on Durability Performance of Building Constructions.

Current edition approved June 10, 1997. Published August 1997. Originally published as E 773 – 81. Last previous edition E 773 – 83 (1995).

[2] *Annual Book of ASTM Standards*, Vol 15.02.

[3] *Annual Book of ASTM Standards*, Vol 04.11.

[4] The apparatus is a modification of the device developed by the Division of Building Research of the National Research Council of Canada. The modifications include two black light lamps for each test specimen. Construction details are available from SIGMA (Sealed Insulating Glass Manufacturer's Association), 111 E. Wacker Drive, Chicago, IL 60601.

[5] The sole source of supply of the apparatus known to the committee at this time is General Electric Co., Nela Park, Cleveland, OH 44112. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

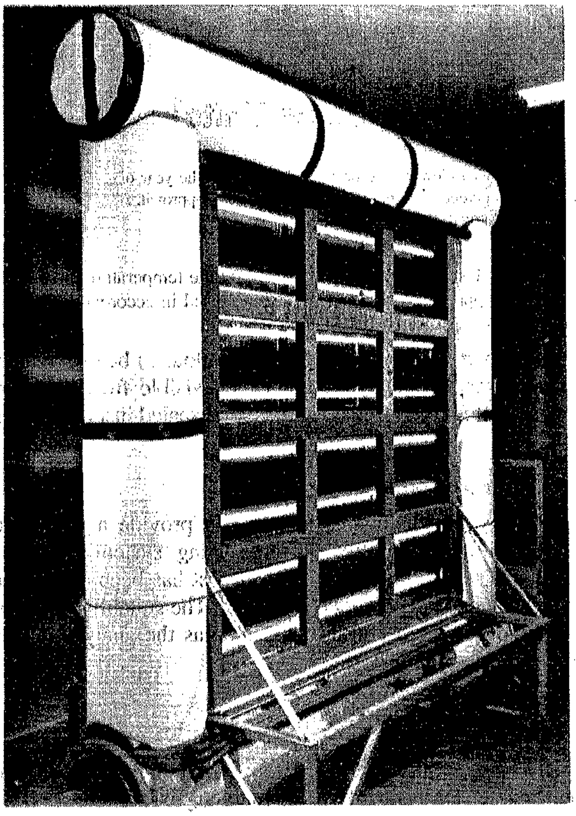


FIG. 1 Typical Accelerated Weather Cycle Test Apparatus

NOTE 3—Rated average life at 3 h per start: 12 000 h. Rated average life at 12 h per start: 18 000 h. Useful length: 1625 mm (64 in.). Wattage: 85 W. Relative black light energy output is 190 % that of F40BL lamp.

6. Test Specimens

6.1 Each test specimen shall measure 356 ± 6 mm by 508 ± 6 mm (14 ± ¼ in. by 20 ± ¼ in.) and shall be composed of two or three panes of clear, tinted or coated annealed, heat-strengthened, tempered or laminated glass.

6.1.1 The thickness of the glass pane shall be nominally 5 mm (3⁄16 in.) or 6 mm (¼ in.).

6.1.2 The airspace or spaces shall be from nominal 6 mm (¼ in.) through 13 mm (½ in.).

6.1.3 Triple pane units where the intermediate airspace divider is a plastic film are acceptable.

6.2 The thickness tolerance of the glass shall conform to Specification C 1036.

6.3 Each specimen shall be permanently and legibly marked with the designation of the manufacturer and the date of fabrication (month and year).

6.4 At least ten specimens of identical component materials and construction shall be submitted.

6.5 During all stages of exposure and storage, the units shall be stated in a vertical position with equal support to all panes and no compression loading.

6.6 Selection of units for testing shall be made at random except for units damaged in transit. Damaged units shall not be tested.

7. Preparation of Test Specimen

7.1 Uncleanable stain or scum may remain on the exterior glass surface of the specimen after the accelerated weathering test. Measures should be taken to have a clear view of the interior glass surface for detection of frost. For example, place a mask of plastic tape[7] 50 by 50 mm (2 by 2 in.) or larger, on the central region of both exterior glass surfaces. Remove the mask for frost point measurement.

7.2 The sealed insulating glass units shall be sealed a minimum of 4 weeks to allow for stabilization before the high humidity exposure in accordance with 8.1 begins. The manufacturer has the option to waive this requirement.

8. Procedure

8.1 *High Humidity Test:*

8.1.1 Expose six specimens in the high-humidity test chamber at 60 ± 3°C (140 ± 5°F) and 95 ± 5 % relative humidity. Arrange the specimens so that each specimen has at least 6-mm (¼-in.) clearance all around the four sides. Protect the high humidity chamber from overheating with a protective device.

8.1.1.1 Equip the high humidity chamber with a continuous temperature recording device placed in an area that gives the average temperature in the chamber.

8.1.2 When the specified time period has been attained, remove the test specimens and determine the frost point in accordance with Test Method E 546. Observe for chemical dew point.

8.2 *Accelerated Weather Cycle Test:*

8.2.1 Place the test specimens that were tested in accordance with 8.1 in the accelerated weather cycle test apparatus. Mount the specimens so that one exterior surface of the specimen is exposed to weathering cycles and the other to room temperature (Note 4). Install all specimens as shown in Fig. 2, taking care that no stress is induced in the test specimens by the method of fastening. The test specimens shall be oriented in the accelerated weather cycle exposure with the number one surface facing the weather changes as it does in normal field exposure. Ensure that the bearing edge and the weathering or exposed side remain the same throughout all testing.

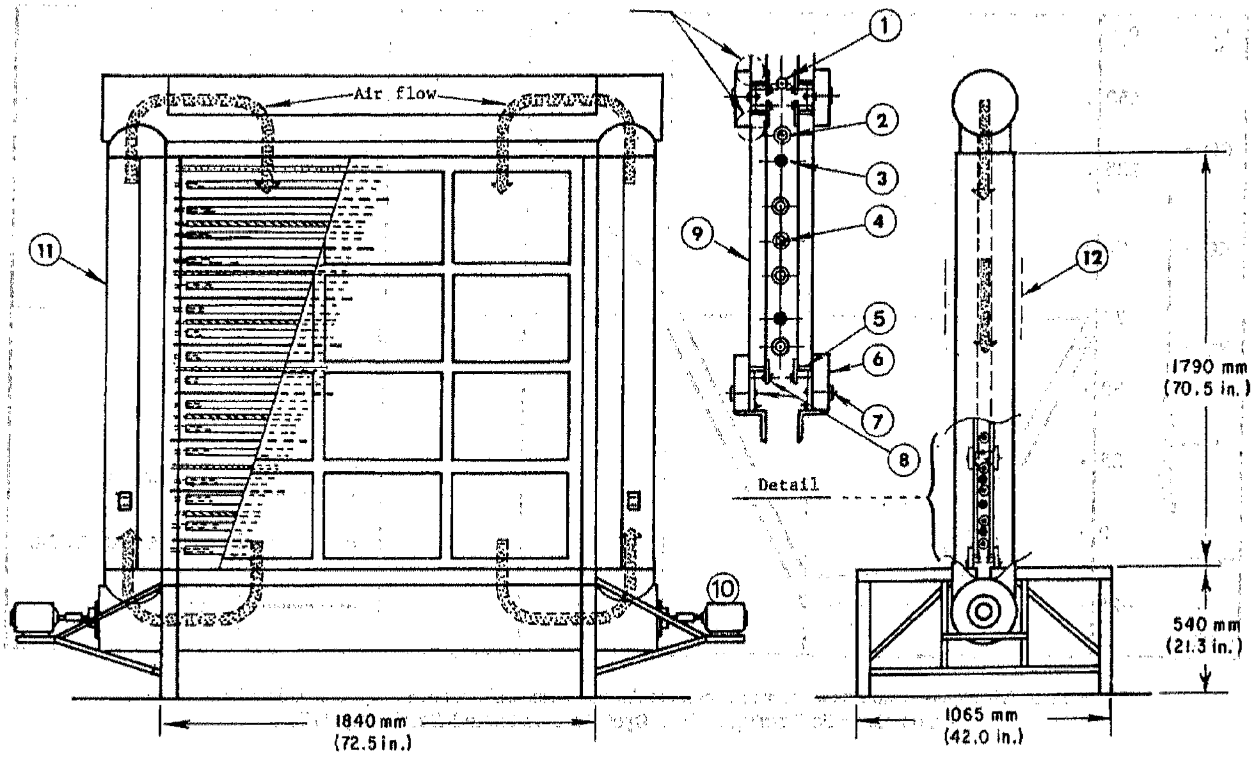
NOTE 4—A temperature in the range from 15 to 30°C (60 to 85°F).

8.2.2 *Cycling*—Each cycle shall be 6 h ± 5 min and composed of the following test condition (see Fig. 4):

---

[6] The sole source of supply of the apparatus known to the committee at this time is the Black-Ray UV Meter with J221 sensor cell available from Ultra-Violet Products, Inc., 5100 Walnut Grove Ave., San Gabriel, CA 91778. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[7] The sole source of supply of the apparatus known to the committee at this time is Scotch Plastic Tape # 471 available from 3M Co., 3M Center, Commercial Office Supply Div., Bldg. 230-3 South-17, St. Paul, MN 55101. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.



Description: 1. Water spray pipe, 2. Cooling coil, 3. Fluorescent black light lamp, F72T12 BL/HO, 4. Heating coil, 5. Rubber pad, 6. Polystyrene insulation, 7. Rubber washer, 8. Clamping device, 9. Test specimen, 10. Fan motor, 11. Air duct, 12. Insulation.

FIG. 2 Schematic Drawing of Typical Accelerated Weather Cycle Test Apparatus

8.2.2.1 During the first 1 h ± 5 min, decrease the temperature from room temperature to − 30 ± 3°C (−20 ± 5°F).

8.2.2.2 Maintain temperature at − 30± 3°C (−20 ± 5°F) for 1 h ± 5 min.

8.2.2.3 Turn on heat and allow temperature to rise from − 30± 3°C (−20 ± 5°F) to room temperature over a period of 1 h ± 5 min.

8.2.2.4 Over a time period of 1 h ± 5 min, start water spray and ultraviolet lamps and control the temperature rise from room temperature to 57 ± 3°C (135 ± 5°F). Turn off water spray after 30 min to allow temperature to continue to rise to 57 ± 3°C.

8.2.2.5 Maintain temperature at 57 ± 3°C (135± 5°F) and continue ultraviolet exposure for a period of 1 h ± 5 min. Humidity remains very high during this hour; consequently, additional water spray is not necessary.

8.2.2.6 Over a period of 1 h ± 5 min, decrease temperature from 57 ± 3°C (135 ± 5°F) to room temperature, and continue ultraviolet exposure. At the end of this period, turn off ultraviolet exposure.

8.2.2.7 Protect the accelerated weathering chamber from overheating and from overcooling with protective devices.

8.2.2.8 Equip the accelerated weather cycle chamber with a continuous temperature recording device placed in an area that gives the average temperature inside the chamber.

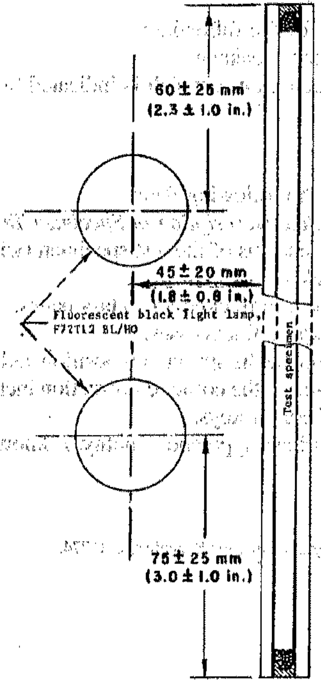
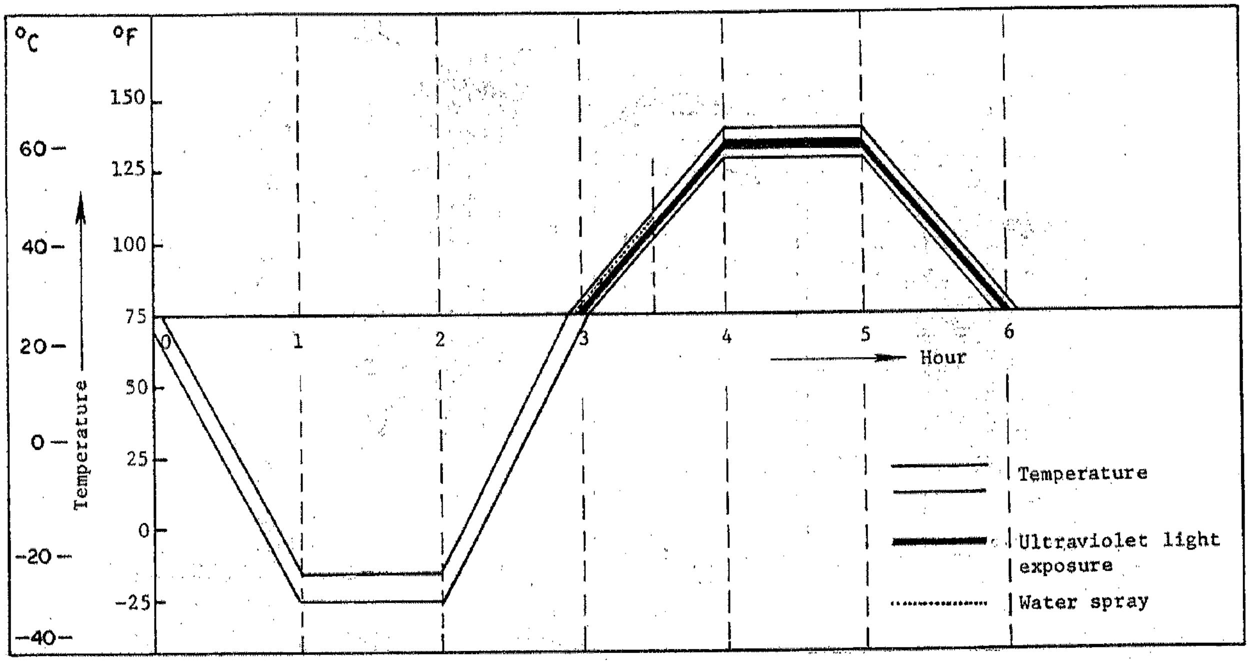


FIG. 3 Location of Fluorescent Black Light Lamp Relative to the Test Specimen



NOTE 1—This figure represents a linear response, but the equipment will not necessarily respond in a linear manner.

FIG. 4 Schematic Drawing of Each Cycle for Accelerated Weather Cycle Test

8.2.3 When the number of cycles prescribed[8] has been attained, remove the specimens and determine the frost point in accordance with Test Method E 546.

9. Observations

9.1 Observe for the following:

9.1.1 Specimen breakage.

9.1.2 Specimen failure, which is indicated by visible water in the cavity.

10. Report

10.1 Report the following data:

10.1.1 *Complete Description of Specimen Tested*:

10.1.1.1 Dimensions of the test specimen (width by height) and overall thickness.

10.1.1.2 Type and thickness of glass panes.

10.1.1.3 Airspace thicknesses.

10.1.1.4 Describe the spacer composition and configuration.

10.1.1.5 Describe the corner construction including the type and number of corner keys.

10.1.1.6 Dessicant type and quantity, if known, and sealant type.

10.1.1.7 Dimensions of sealant.

10.1.1.8 Manufacturer and manufactured date (month, if known, and year).

10.1.2 *Duration of Test*:

10.1.2.1 Duration of high humidity test described in 8.1 (number of days or weeks).

10.1.2.2 Duration of accelerated weather cycle test described in 8.2 (number of cycles, days or weeks).

10.1.3 Chemical dew point, if observed.

10.1.4 Specimen breakage, if observed.

10.1.5 Massive specimen failure, if observed.

11. Precision and Bias

11.1 *Precision*—The precision of the procedures in Test Method E 773 for measuring accelerated weatherability is being determined.

11.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedures in Test Method E 773 for accelerated weatherability, bias has not been determined.

12. Keywords

12.1 insulating glass units; sealed insulating glass units

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[8] For insulating glass units, see Specification E 774.

CERTIFICATE

By Authority Of
THE UNITED STATES OF AMERICA
Legally Binding Document

By the Authority Vested By Part 5 of the United States Code § 552(a) and Part 1 of the Code of Regulations § 51 the attached document has been duly INCORPORATED BY REFERENCE and shall be considered legally binding upon all citizens and residents of the United States of America. *HEED THIS NOTICE*: Criminal penalties may apply for noncompliance.

◆

Document Name: ASTM E774: Standard Specifications for Sealed Insulatin Glass Units

CFR Section(s): 24 CFR 3280.403(d)(2)

Standards Body: American Society for Testing and Materials

APPROVED

Official Incorporator:
THE EXECUTIVE DIRECTOR
OFFICE OF THE FEDERAL REGISTER
WASHINGTON, D.C.

Designation: E 774 – 97 An American National Standard

Standard Specification for the Classification of the Durability of Sealed Insulating Glass Units[1]

This standard is issued under the fixed designation E 774; 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 specification is applicable to those sealed insulating glass units, with one or two airspaces.

1.2 The classification of test specimens is based on frost or chemical dew point after the specified test duration(s).

1.3 Qualification under this specification is intended to provide a basis for the classification of the durability of sealed insulating glass units. SIGMA field correlation studies started in 1980 show that units that have achieved a Level A classification have less than 1 % field failure rate in ten years provided the glazing system weeps water away from contact with the unit edge seal. Class C and CB tested units have significantly higher field failure rates in ten years. SIGMA continues to gather correlation data.[2]

1.4 This specification is not applicable to units that are constructed from vision materials other than glass.

1.5 This specification does not cover other physical requirements such as appearance, thermophysical properties, heat and light transmission, and glass displacement.

NOTE 1—Sealed insulating glass units classified according to this specification are not necessarily suitable for structurally glazed applications. Factors such as sealant longevity to long term direct ultraviolet light exposure and sealant tensile strength must be reviewed for these applications.

2. Referenced Documents

2.1 *ASTM Standards:*

E 546 Test Method for Frost Point of Sealed Insulating Glass Units[3]

E 773 Test Method for Accelerated Weathering of Sealed Insulating Glass Units[3]

E 1887 Test Method for Fog Determination[3]

3. Terminology

3.1 Definition:

3.1.1 *sealed insulating glass unit*—a preassembled unit, comprising sealed panes of glass separated by dehydrated space(s), intended for vision areas of buildings. The unit is normally used for windows, window walls, picture windows, sliding doors, patio doors, or other types of windows or doors.

4. Classification

4.1 Sealed insulating glass units tested in accordance with this specification shall be classified into one of three classes based on response to the tests and qualification requirements as shown in Table 1.

5. Performance Requirements

5.1 *Class C*—There shall be no frost or chemical dew point of each specimen when measured at a temperature of −34°C (−30°F) in accordance with Test Method E 546.

5.2 *Class B*—There shall be no frost or chemical dew point when measured at a temperature of −29°C (−20°F) in accordance with Test Method E 546.

5.3 *Class A*—There shall be no frost or chemical dew point when measured at a temperature of −29°C (−20°F) in accordance with Test Method E 546.

5.4 *Fog*—No fog shall be visible after testing in accordance with Test Method E 1887.

6. Test Specimens

6.1 Specimen design and construction for accelerated weathering tests shall be established by Test Method E 773.

7. Test Methods

7.1 Classify the sealed insulating glass units by following Test Method E 773 in accordance with Table 1 of this specification.

7.1.1 *Accelerated Weathering Test*:

7.1.1.1 Test six randomly selected test specimens for durations as shown in Table 1, Class C in accordance with Test Method E 773.

7.1.1.2 If the test specimens qualify for Class C as described in Section 5 of this specification, they may be tested further for durations as shown in Table 1, Class B. Place the specimens in the test racks so that the bearing edge and the weathering or exposed side remain the same as tested in previous class.

7.1.1.3 If the test specimens qualify for Class B as described in Section 5 of this specification, they may be tested further for durations as shown in Table 1, Class A.

7.1.1.4 Breakage of only two specimens is permitted

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[1] This specification is under the jurisdiction of ASTM Committee E-6 on Performance of Buildings and is the direct responsibility of Subcommittee E06.22 on Durability Performance of Building Constructions.

Current edition approved June 10, 1997. Published August 1997. Originally published as E 774 – 81. Last previous edition E 774 – 92.

[2] Available from SIGMA, 111 East Wacker Drive, Suite 600, Chicago, IL 60601.

[3] *Annual Book of ASTM Standards*, Vol 04.11.

TABLE 1 **Classification and Physical Requirements for Seal Durability Test Method E 773**

Classification of Specimen	Record Initial Frost Point and Chemical Dew Point	Accelerated Weathering Test			
		Number of Specimens	Duration of Test		Final Qualification of Frost Point and Chemical Dew Point
			High Humidity Test,^{A} (days)	Accelerated Weather Cycle Test,^{B} cycles	
Class C		6 each	14	140	lower than – 34°C (–30°F)
Class B		6 each	14	56	lower than – 29°C (–20°F)
Class A		6 each	14	56	lower than – 29°C (–20°F)

Fogging Test		
Number of Specimens	Duration of Test, days	Qualification
2 each	7	no visible fog

^{A} See Procedure section of Test Method E 773.
^{B} See Procedure section of Test Method E 773.

throughout the test. Replace any broken specimen with one from the same lot, and test from the beginning.

NOTE 2—Breakage due to laboratory handling, that is dropped units, is not considered to be test breakage. Laboratory broken units shall be replaced and retested from the beginning.

7.1.1.5 If third specimen breakage occurs during the test for any reason, do not qualify this set of test specimens for that tested class.

7.1.1.6 If any specimen has filled with water or shows visible condensation in the air space, do not qualify this set of test specimens for that tested class except as permitted in 7.1.1.4 (that is, breakage).

7.1.2 *Fogging Test:*

7.1.2.1 Select two specimens as shown in Table 1.

7.1.2.2 Test the two test specimens for durations as shown in Table 1, Class C in accordance with Test Method E 1887.

7.2 The test durations as shown in Table 1 are net testing periods. If the test is interrupted for any reason, the remaining portions shall be completed to qualify for the testing class.

NOTE 3—It is suggested that extra units be submitted. Twelve units have been shown to be adequate in many cases.

8. Report

8.1 Report the following information:

8.1.1 Detailed description of test specimen, test durations, and all reportable test data of Test Methods E 773 and E 1887.

8.1.2 Classes for which the test specimens qualified.

9. Acceptance or Rejection

9.1 When all test specimens have met the requirements as described in Section 5 for any particular class, this set of test specimens shall be accepted for that tested class.

9.2 If any specimen fails to meet the requirements as described in Section 5 for any particular class, this set of specimens shall be rejected for that tested class.

10. Keywords

10.1 insulating glass units; sealed insulating glass units

APPENDIX

(Nonmandatory Information)

X1. TEST DATA GATHERING

FIG. X1.1 Test Report for Specification E 774

Date ________
Manufacturer ________ Ref. No. ________ Testing Lab. ________ Ref. No. ________
Address ________ Address ________
Attention ________ Tel. ________ Attention ________ Tel. ________
Description of Test Specimen:
Size (width by height) ________ Type and amount of desiccator ________
Thickness of glass ________ Type of sealant(s) ________
Thickness of air space ________ Other features (band, barrier coat, etc.) ________
Type of spacer ________ Manufactured date (month and year) ________
Test Results

Specimen No.	Class C					Class B			Class A		
	Initial Frost Point	Fogging Test, days	High-Humidity Test,[A] days	Accelerated Weather Cycle Test,[A] cycles	Remark	High-Humidity Test,[A] days	Accelerated Weather Cycle Test,[A] cycles	Remark	High-Humidity Test,[A] days	Accelerated Weather Cycle Test,[A] cycles	Remark
		7	14	140		14	56		14	56	
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											

[A] Actual frost/chemical dew point, or pass/fail.