



# Standard Test Method for Vapor Pressure of Petroleum Products (Automatic Method)<sup>1</sup>

This standard is issued under the fixed designation D 5190; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope \*

1.1 This test method covers the determination of the total pressure of air-containing, volatile, petroleum products. This test method is suitable for testing samples with boiling points above 0°C (32°F) that exert a vapor pressure between 7 and 172 kPa (1 and 25 psi) at 37.8°C (100°F) at a vapor-to-liquid ratio of 4:1. This test method is suitable for testing gasoline samples that contain oxygenates. No account is made of dissolved water in the sample.

1.1.1 Some gasoline-oxygenate blends may show a haze when cooled to 0 to 1°C. If a haze is observed in 8.5, it shall be indicated in the reporting of results. The precision and bias statements for hazy samples have not been determined (see Note 8).

1.2 This test method is suitable for the calculation of a dry vapor pressure equivalent (DVPE) by means of a correlation equation (see 13.1). The calculated DVPE very closely approximates the dry vapor pressure that would be obtained on the same material when tested in accordance with Test Method D 4953.

1.3 The values stated in SI units are to be regarded as the standard. The inch-pound units given in parentheses are provided for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see 7.3 through 7.5 and 9.2.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>2</sup>

D 4953 Test Method for Vapor Pressure of Gasoline and Gasoline-Oxygenate Blends (Dry Method)<sup>3</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.08 on Volatility.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.03.

D 5191 Test Method for Vapor Pressure of Petroleum Products (Mini Method)<sup>3</sup>

2.2 ASTM Adjuncts:

Detailed Drawings for Automatic Vapor Pressure Instrument<sup>4</sup>

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *total pressure, n*—the observed pressure measured in the experiment, that is, the sum of the partial pressure of the sample and the partial pressure of the dissolved air.

3.1.2 *dry vapor pressure equivalent (DVPE), n*—a value calculated by a correlation equation (see 13.1) from the total pressure.

3.1.2.1 *Discussion*—The DVPE is expected to be equivalent to the value obtained on the sample by Test Method D 4953, Procedure A.

## 4. Summary of Test Method

4.1 The chilled sample cup of the automatic vapor pressure instrument is filled with chilled sample and is coupled to the instrument inlet fitting. The sample is then automatically forced from the sample chamber to the expansion chamber where it is held until thermal equilibrium at 37.8°C (100°F) is reached. In this process the sample is expanded to five times its volume (4:1 vapor-to-liquid ratio). The vapor pressure is measured by a pressure transducer.

4.2 The measured vapor pressure is automatically converted to a DVPE value by the instrument. A correction to this value is necessary to account for the observed bias between the test result and that obtained by Test Method D 4953.

## 5. Significance and Use

5.1 Vapor pressure is an important physical property of volatile liquids.

5.2 The vapor pressure of gasoline and gasoline-oxygenate blends is regulated by various government agencies.

5.3 Specifications for volatile petroleum products generally include vapor pressure limits to ensure products of suitable volatility performance.

<sup>4</sup> Available from ASTM Headquarters. Order PCN 12-500140–12.

\*A Summary of Changes section appears at the end of this standard.

5.4 This test method is more precise than Test Method D 4953.

## 6. Apparatus

6.1 *Automatic Vapor Pressure Instrument*,<sup>5</sup> the essential features describing the sample flow and operation of the automatic vapor pressure instrument is provided in Annex A1. Critical elements of the apparatus are included as follows:

6.1.1 *Pressure Transducer*, capable of operating in the range from 0 to 172 kPa (0 to 25 psi) with the resolution of 0.1 kPa (0.01 psi) and a minimum accuracy of  $\pm 0.7$  kPa ( $\pm 0.10$  psi).

6.1.2 *Thermostatically Controlled Heater*, capable of maintaining an oil bath surrounding the test chambers at  $37.8 \pm 0.1^\circ\text{C}$  ( $100 \pm 0.2^\circ\text{F}$ ) for the duration of the test.

6.1.3 *Sample Cup*, capable of holding up to 125 mL.

6.2 *Iced-Water Bath or Air Bath*, for chilling the test samples and sample cup to temperatures between 0 to  $1^\circ\text{C}$  (32 to  $34^\circ\text{F}$ ).

## 7. Reagents and Materials

7.1 *Purity of Reagents*—Chemicals of at least 98 % purity shall be used in the calibration procedure (see Section 10). Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.<sup>6</sup> Lower purities may be used, provided it is first ascertained that the reagent is of sufficient purity to permit its use without lessening the accuracy of the determination.

NOTE 1—Although higher purity chemicals in the 99 + % range are preferred for use in calibrations, the precision and bias statements (see Section 15) were derived with the minimum purity level stated in 7.1.

7.2 *2,2-Dimethylbutane*.

7.3 *n-Hexane*, (**Warning**—2,2-Dimethylbutane, *n*-hexane is extremely flammable, harmful if inhaled. Skin irritant on repeated contact. Aspiration hazard).

7.4 *n-Pentane*, (**Warning**—2,2-Dimethylbutane, *n*-pentane is extremely flammable, harmful if inhaled. Skin irritant on repeated contact. Aspiration hazard).

7.5 *Toluene*, (**Warning**—2,2-Dimethylbutane, toluene is extremely flammable, harmful if inhaled. Skin irritant on repeated contact. Aspiration hazard).

## 8. Sampling

8.1 *General Procedures*:

8.1.1 The extreme sensitivity of vapor pressure measurements to losses through evaporation and the resulting change in composition is such as to require the utmost precaution and the most meticulous care in the drawing and handling of samples.

8.1.2 Obtain a sample and test specimen in accordance with Practice D 4057, except do not use “Sampling by Water Displacement” for fuels containing oxygenates. Use a 1-L (1-qt) size container filled between 70 and 80 % with the sample.

NOTE 2—The present precision statement was derived using the samples in 1-L (1-qt) containers. However, the samples in containers of other sizes, as prescribed in Practice D 4057 can be used, with the same ullage requirement, if it is recognized that the precision can be affected.

8.1.3 In the case of referee testing, the 1-L (1-qt) sample container is mandatory.

8.1.4 Perform the vapor pressure determination on the first test specimen withdrawn from a sample container. Do not use the remaining sample in the container for a second vapor pressure determination. If a second determination is necessary, obtain a new sample.

8.1.5 Protect the samples from excessive temperatures prior to testing. This can be accomplished by storage in an appropriate ice bath or refrigerator.

8.1.6 Do not test samples stored in leaky containers. If leaks are detected, discard and obtain a new sample.

8.2 *Sampling Handling Temperature*—Cool the sample container and contents in an ice bath or refrigerator to between 0 and  $1^\circ\text{C}$  (32 and  $34^\circ\text{F}$ ) prior to opening the sample container. Allow sufficient time to reach this temperature. Verify the sample temperature by direct measurement of the temperature of a similar liquid in a similar container placed in the cooling bath or refrigerator at the same time as the sample.

8.3 *Verification of Sample Container Filling*—With the sample at a temperature of 0 to  $1^\circ\text{C}$ , take the container from the cooling bath or refrigerator and wipe dry with absorbent material. If the container is not transparent, unseal it and using a suitable gage, confirm that the sample volume equals 70 to 80 % of the container capacity (see Note 3). If the sample is contained in a transparent glass container, verify that the container is 70 to 80 % full by suitable means (see Note 3).

NOTE 3—For non-transparent containers, one way to confirm that the sample volume equals 70 to 80 % of the container capacity is to use a dipstick that has been pre-marked to indicate the 70 and 80 % container capacities. The dipstick should be of such material that it shows wetting after being immersed and withdrawn from the sample. To confirm the sample volume, insert the dipstick into the sample container so that it touches the bottom of the container at a perpendicular angle, before removing the dipstick. For transparent containers, using a marked ruler or comparing the sample container to a like container that has the 70 and 80 % levels clearly marked, has been found suitable.

8.3.1 Discard the sample if the container is filled to less than 70 volume % of the container capacity.

8.3.2 If the container is more than 80 volume % full, pour out enough sample to bring the container contents within 70 to 80 volume % range. Do not return any sample to the container once it has been withdrawn.

8.3.3 Reseal the container, if necessary, and return the sample container to the cooling bath or refrigerator.

8.4 *Air Saturation of the Sample in the Sample Container*:

8.4.1 *Transparent Containers Only*—Since 8.3 does not require that the sample container be opened to verify the sample capacity, it is necessary to unseal the cap momentarily before resealing it, so that samples in transparent containers are

<sup>5</sup> The following instrument has been found suitable by interlaboratory cooperative testing: Vapor Pressure Instrument, available from Southwest Research Institute, San Antonio, Texas.

<sup>6</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeial and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

treated the same as samples in non-transparent containers.

8.4.2 With the sample again at a temperature of 0 to 1°C, take the container from the cooling bath or refrigerator, wipe it dry with an absorbent material, remove the cap momentarily, taking care that no water enters, reseal and shake vigorously. Return it to the bath or refrigerator for a minimum of 2 min.

8.4.3 Repeat 8.4.2 twice more. Return the sample to the bath or refrigerator until the beginning of the procedure.

8.5 *Verification of Single Phase Samples*—After drawing a test specimen into the sample cup and coupling the cup to the instrument for analysis, check the remaining sample for phase separation. If the sample is contained in a glass container, this observation can be made prior to sample transfer. If the sample is contained in a non-transparent container, mix the sample thoroughly and immediately pour a portion of the remaining sample into a clear glass container and observe for evidence of phase separation. A hazy appearance is to be carefully distinguished from separation into two distinct phases. The hazy appearance shall not be considered grounds for rejection of the fuel. If a second phase is observed, discard the test and the sample. Hazy samples may be analyzed (see Report section).

## 9. Preparation of Apparatus

9.1 Prepare the automatic vapor pressure instrument for operation in accordance with the manufacturer's instructions.

9.2 Clean and dry the sample cup prior to use. (**Warning**—Do not analyze a water saturated sample. If water is accidentally introduced into the instrument, analyze a dry sample six to ten times until all the water has been flushed from the instrument and a repeatability of  $\pm 1.4$  kPa (0.20 psi) is obtained for duplicate runs.)

9.3 Chill the stoppered, dry sample cup to between 0 and 1°C (32 and 34°F) in a refrigerator or ice bath before transferring the sample into the cup. Avoid water contamination of the sample cup by sealing the sample cup during the cooling process.

9.4 Prior to starting the measurement, check that the temperature of the test chamber is within the required range specified by the manufacturer of the instrument.

## 10. Calibration

### 10.1 Pressure Transducer:

10.1.1 Calibrate the pressure transducer at least every 30 days or when needed as indicated from the performance checks. The calibration of the transducer is accomplished using three reference materials to cover the range above and below 34 kPa (5.0 psi).

NOTE 4—The instrument manufacturer provides an alternative calibration procedure using two reference points, zero pressure (<0.1 kPa) and the ambient barometric pressure. However, since this procedure was not included in the interlaboratory program, the precision and bias can be affected by its use.

10.1.2 Load *n*-hexane into the instrument, obtain a vapor pressure reading and then adjust the zero potentiometer for the transducer to obtain a calibration value of  $5.00 \pm -0.02$  on the digital meter display.

NOTE 5—The target calibration values used in this section are specific to the automatic vapor pressure instrument<sup>5</sup> evaluated in the 1988

interlaboratory cooperative program.<sup>7</sup> These calibration values do not necessarily correspond to the total vapor pressures or the dry vapor pressures (see Test Method D 4953) reported for the reference calibration materials, but rather are values that the instrument manufacturer suggests using to produce a dry vapor pressure equivalent reading on the digital display.

10.1.3 Load *n*-pentane into the instrument, obtain a pressure reading on the digital meter, and then adjust the transducer span potentiometer to achieve a value of  $15.40 \pm -0.05$  on the digital meter.

10.1.4 Repeat 10.1.2 and 10.1.3 until the appropriate calibration values are displayed without making further adjustments.

10.1.5 Load the instrument with 2,2-dimethylbutane and obtain a pressure reading. If the digital display reads  $9.90 \pm 0.1$ , then the instrument is calibrated; if not, then repeat the above procedure until a satisfactory calibration is achieved.

10.1.6 For calibration of the range below 34 kPa (5.0 psi), perform the steps in 10.1.2 to 10.1.4, replacing *n*-hexane (34 kPa) in step 10.1.2 with toluene (7 kPa), and replacing *n*-pentane (106 kPa) in step 10.1.3 with *n*-hexane (34 kPa).

10.2 *Temperature*—At least every six months, check the calibration of the thermometer used in the thermostatically controlled bath against a National Institute of Standards and Technology (NIST) traceable thermometer and check the capability of the bath thermostat control to maintain a temperature of  $37.8 \pm 0.1^\circ\text{C}$  ( $100 \pm 0.2^\circ\text{F}$ ) throughout the measurement period. Take corrective actions when the thermometer and thermostat exceed the limits stated.

## 11. Quality Control Checks

11.1 Use a verification fluid with known volatility as an independent check against the instrument calibration each day the instrument is in use. For pure compounds (see 7.1 and Note 1), multiple test specimens may be taken from the same container over time, provided the pure compound is air saturated according to the procedure given in 8.4 and the spent test specimens are not re-used, in whole or in part. Record the dry vapor pressure equivalent value, and compare this to the statistical value of the control sample from your laboratory. If the difference exceeds your control limits, check the calibration of the instrument.

11.2 Some possible materials and their corresponding vapor pressures, as found in ASTM DS4B,<sup>8</sup> include: cyclopentane, 68.3 kPa (9.91 psi); 2,2-dimethylbutane 68.0 kPa (9.86 psi); 2,3-dimethylbutane 51.1 kPa (7.41 psi); 2-methylpentane 46.7 kPa (6.77 psi); and toluene 7.1 kPa (1.03 psi).<sup>9</sup>

NOTE 6—It is recommended that at least one type of control sample used in 11.1 be representative of the fuel(s) regularly tested by the laboratory. The total vapor pressure measurement process (including operator technique) can be checked periodically by performing this test method on previously prepared samples from one batch of product, as per

<sup>7</sup> Supporting data are available from ASTM Headquarters. Request RR:D02-1260.

<sup>8</sup> "Physical Constants of Hydrocarbon and Non-Hydrocarbon Materials," available from ASTM Headquarters. Order ADJPCN 28-003092-12.

<sup>9</sup> The vapor pressure values cited were obtained from Phillips Petroleum Company, Bartlesville, Oklahoma, or ASTM DS 4B Physical Constants of Hydrocarbon and Non-Hydrocarbon Compounds.

procedure described in 8.1.2. Samples should be stored in an environment suitable for long term storage without sample degradation. Analysis of result(s) from these quality control samples can be carried out using control chart techniques<sup>10</sup> or other statistically equivalent techniques.

**12. Procedure**

12.1 *Sample Transfer*—Remove the sample from the cooling bath or refrigerator, dry the exterior of the container with absorbent material, uncap, and insert a chilled transfer tube apparatus (see Fig. 1). Quickly take the chilled sample cup and place it, in an inverted position, over the sample delivery tube of the transfer apparatus. Invert the entire system rapidly so that the sample cup is upright with the end of the delivery tube touching the bottom of the sample cup. Fill the sample cup to overflowing. Withdraw the delivery tube from the sample cup while allowing the sample to continue flowing up to the moment of complete withdrawal.

NOTE 7—**Precaution:** In addition to other precautions make provisions for suitable restraint (for example, catch pan) and disposal of the overflowing or spilled gasoline to avoid fire hazard.

12.2 Quickly couple the sample cup to the instrument and start the analysis in accordance with the manufacturer’s instructions for operation of the instrument. The total time between opening the chilled sample container and securing the sample cup to the instrument shall not exceed 1 min.

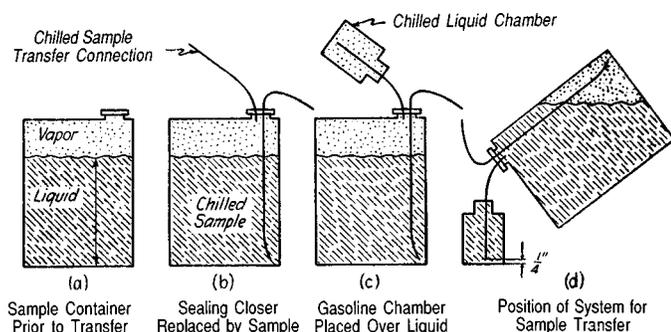
12.3 At the completion of the test, record the uncorrected dry vapor pressure reading from the digital meter to the nearest 0.1 kPa (0.01 psi). If the instrument does not automatically calculate the DVPE, record the uncorrected vapor pressure reading and calculate the DVPE using Eq 1 (see 13.1).

**13. Calculation**

13.1 Calculate a DVPE, using the following equation. This corrects the instrument reading for the relative bias found in the 1991 interlaboratory cooperative test program (see Note 10) between the dry vapor pressure measured in accordance with Test Method D 4953, Procedure A and this test method:

$$DVPE = (0.954 X) + A \tag{1}$$

<sup>10</sup> Reference ASTM MNL 7, *Manual on Presentation of Data and Control Chart Analysis*, Sixth Edition, Section 3: Control Charts for Individuals.



**FIG. 1 Simplified Sketches Outlining Method Transferring Sample to Liquid Chamber from Open-Type Containers**

where:

- X = measured total vapor pressure, in units consistent with A, and
- A = 1.94 kPa (0.281 psi).

**14. Report**

14.1 Report the corrected dry vapor pressure equivalent pressure to the nearest 0.1 kPa (0.01 psi) without reference to temperature.

14.2 If the sample was observed to be hazy in 8.5, report the test result as in 14.1, followed by the letter “H”.

NOTE 8—The precision and bias statements have not been determined for hazy samples, since these types of samples have not been evaluated as part of an interlaboratory study.

NOTE 9—The inclusion of the letter “H” in 14.2 is intended to alert the data recipient that the sample analyzed was hazy. In the event a laboratory has a computer system that is incapable of reporting alphanumeric results according to the requirements in 14.2, it is permissible for the laboratory to report the result obtained as in 14.1, along with a statement or annotation that clearly conveys to the data recipient that the sample analyzed was hazy.

**15. Precision and Bias**

15.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory test results is as follows: The following precision data were developed in a 1991 interlaboratory cooperative test program. Participants analyzed sample sets comprised of blind duplicates of 14 types of hydrocarbons and hydrocarbon-oxygenate blends. The oxygenate content (MTBE, ethanol, and methanol) ranged from 0 to 15 % by volume nominal and the vapor pressure ranged from 14 to 100 kPa (2 to 15 psi) nominal. A total of 60 laboratories participated. Some participants performed more than one test method, using separate sample sets for each. Twenty-six samples sets were tested by Test Method D 4953, 13 by this test method, and 27 by Test Method D 5191. In addition, six sets were tested by modified D 5190 and 13 by modified Test Method D 5191.<sup>11</sup>

15.1.1 *Repeatability*—The difference between successive test results obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following value only in one case in twenty:

$$2.48 \text{ kPa (0.36 psi)}$$

15.1.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, exceed the following value only in one case in twenty:

$$3.45 \text{ kPa (0.50 psi)}$$

15.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedures in this test method, bias cannot be determined.

15.2.1 A statistically significant relative bias was observed in the 1991 interlaboratory cooperative test program described

<sup>11</sup> The results of this test program are filed at ASTM Headquarters. Request RR:D02-1286.

in 15.1 between the total pressure obtained using this test method and the dry vapor pressure obtained from using Test Method D 4953, Procedure A. This bias can be corrected by applying Eq 1, as described in Section 13.

**16. Keywords**

16.1 automated vapor pressure measurement; dry vapor pressure; gasoline; hydrocarbon-oxygenate blends; petroleum products; vapor pressure

**ANNEX**

**(Mandatory Information)**

**A1. ESSENTIAL COMPONENTS AND FUNCTION OF AN AUTOMATIC VAPOR PRESSURE INSTRUMENT**

A1.1 The essential components of an automatic vapor pressure instrument are indicated in Fig. A1.1. The instrument consists of a system of valves, tubes, and expansion chambers that automatically loads a sample into a pressure chamber and then expands the chamber volume by five times. A pressure transducer measures the resulting vapor pressure of the sample.

*A1.2 Operations Sequence for Loading Sample:*

A1.2.1 Depression of the start switch provides the impulse to start the analysis cycle. A timing board circuit operates to produce the required analysis program. Refer to Fig. A1.1 for the identification of the various part numbers (indicated in parentheses) that are essential to the operation.

A1.2.2 After initiation, air valve (5) opens to apply 96.5 kPa (13.8 psi) to the sample cup (27).

A1.2.3 Drain valve (6) opens for 1 s to permit 5 to 15 mL of sample to flow to the drain.

A1.2.4 Pneumatic valve (1) is normally open in the C-A position, maintaining pressure on the top of the air cylinder (15) to keep the piston assembly (22) in the down stroke position. In this position, pneumatic valve (2) is normally closed in the B-A position.

A1.2.5 Pneumatic valves (1), (2), (3) and (4) are electrically tied together. When power is applied to these valves, pneumatic valve (1) closes to C-A position, valve (2) opens to B-A position, sample valve (3) and flush valve (4) open simultaneously. Pneumatic pressure applied to cylinder piston (15) through valve (2), with relief through valve (1), forces the piston up (this is the fill stroke). As the piston moves up, 10 to 15 mL of sample is drawn into sample cylinder (24), and the residue from the previous sample is evacuated from the expansion chamber (26) through flush valve (4). This fill stroke requires about 10 s.

A1.2.6 Power is removed from pneumatic valve (2) that closes position B-A. Pneumatic valve (1) is open to C-A. Sample valve (3) and flush valve (4) and close and transfer valve (7) opens. Pneumatic pressure applied through valve (1), with relief through valve (2), forces the piston down. Liquid is forced from the sample chamber through transfer valve (7) into the expansion chamber. Stroke time is about 10 s.

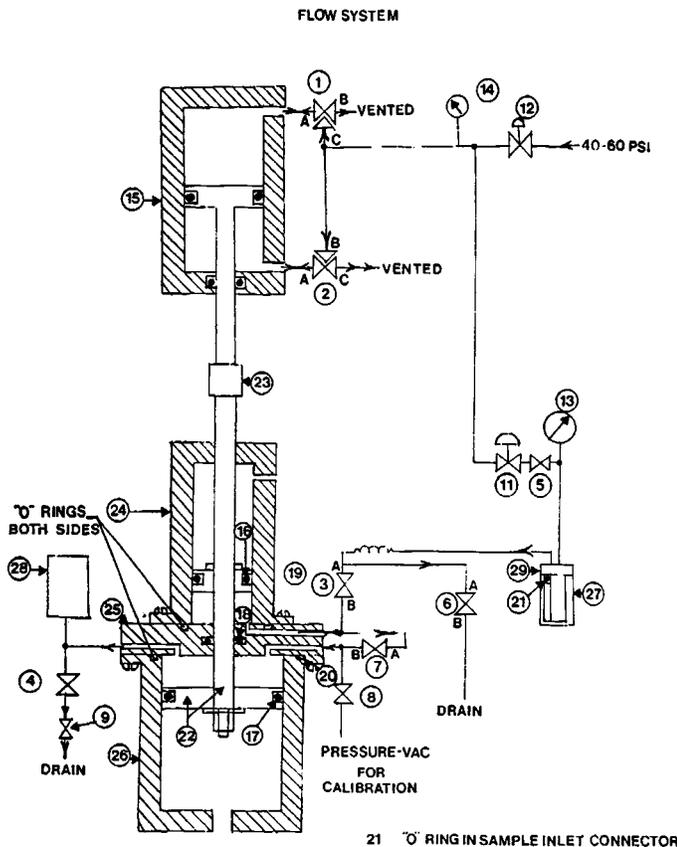
A1.2.7 Steps A1.2.5 and A1.2.6 are repeated three times for a total of four samplings, each taking 10 to 15 mL of the sample. The first three samplings are used to flush the system clean. The fourth sample is held in the expansion chamber to thermal equilibrium.

A1.2.8 At the end of the fourth expansion stroke, transfer valve (7) closes. The piston is retained in the down position by pneumatic pressure through valve (1). Sample cup drain valve (6) opens, and the sample remaining in the cup (27) is forced to drain.

A1.2.9 Conditions remain static for about 3.5 min to permit the expanded sample in the chamber to attain thermal equilibrium.

*A1.3 Measurement Cycle:*

A1.3.1 Approximately 45 s before the end of the cycle, the digital display meter for the transducer is freed, permitting it to display the vapor pressure of the sample as received from the pressure transducer (28). Air valve (5) and sample cup drain valve (6) close before the display meter locks for the next run.



**FIG. A1.1 Flow System for Automatic Vapor Pressure Instrument**

A1.3.2 At the end of the analysis cycle, the dry vapor pressure equivalent is automatically calculated and displayed.

### SUMMARY OF CHANGES

Committee D02.08 has identified the location of selected changes to this standard since the last issue (D 5190–99) that may impact the use of this standard.

(J) Paragraph 11.2, removed cyclohexane as a quality control check since it is solid at the test temperature of 0°C.

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