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Bubble-Point Test Method

1. SCOPE:

This test method describes a procedure for measuring the largest pore or hole in a filter or similar fluid-permeable porous structure. A standard referee test method for precise determination or resolution of disputes is specified. A simpler inspection test procedure for quality assurance "go-no-go" measurement is also given. Bubble-point testing physics, analysis of bubble-point test data, and correlation with other methods of pore size determination are separately discussed in the appendix.

2. APPLICABLE DOCUMENTS:

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

2.1 U.S. Government Publications:

Available from DODSSP, Subscription Services Desk, Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094.

MIL-H-5606	Hydraulic Fluid, Petroleum Base; Aircraft; Missile and Ordnance
MIL-PRF-5624	Turbine Fuel, Aviation, Grades JP-4, JP-5, JP5/JP-8 ST
MIL-E-51454	Ethyl Alcohol, (Ethanol)

3. METHOD OUTLINE:

The filter is immersed in a test liquid which wets and saturates the filter pore structure. Gas pressure is applied to one side of the porous wall so that the liquid phase which wets the pores is displaced by the gas. The gas pressure is slowly increased until the first steady stream of gas bubbles is observed to emit from a point on the porous surface. The measured gas pressure required to form the "first bubble" is essentially equal to the pressure force which is in equilibrium with the surface tension force at the largest pore. This bubble-point pressure is a relative measure of the pore size after appropriate corrections for immersion depth and test liquid surface tension have been made.

4. REFEREE TEST:

Capillary pressure or "bubble-point" tests have long been used to measure the largest pore and characterize the pore size distribution of various porous materials. Many different methods of testing and analysis have been developed. No bubble-point test measures actual pore size, but only allows correlation of the measured capillary pressure with some dimensional characteristic of the pore structure. Rationalization of bubble-point test data depends on detailed definition of the test method, analysis and correlation employed. Therefore, a standard referee test is necessary to assure common interpretation of data and understanding of specifications.

4.1 Test Equipment:

Suitable bubble-point test equipment includes a source of clean compressed air or nitrogen with provisions for regulating and measuring the gas pressure. A test liquid container and appropriate fixtures for sealing and holding the test element are also required. Further provisions for maintaining system cleanliness may be desirable, especially for Clean Room operations, but they are not generally required for ordinary testing. A suitable apparatus for typical filter elements is shown schematically in Figure 1 and requires the following components or their functional equivalent.

4.1.1 Shut-off valve (optional) to allow disconnecting and servicing other components (1).

4.1.2 Water-oil separator/filter or other provision to insure clean compressed air or nitrogen gas (2).

4.1.3 Primary pressure regulator to reduce gas pressure below the maximum inlet pressure of the low pressure regulator (3).

4.1.4 Secondary precision low pressure regulator to (0 to 50 in of water pressure or similar range) for final gas pressure control (4).

4.1.5 Three-way PTFE plug valve (optional) to facilitate venting of test elements (5).

4.1.6 Manometer, preferably well-type, or calibrated pressure gage to read gas pressure with a precision of at least 0.1 in of water pressure (2.54 mm) (6).

4.1.7 Tank or similar container for holding test liquid and allowing observation of bubbles from test element.

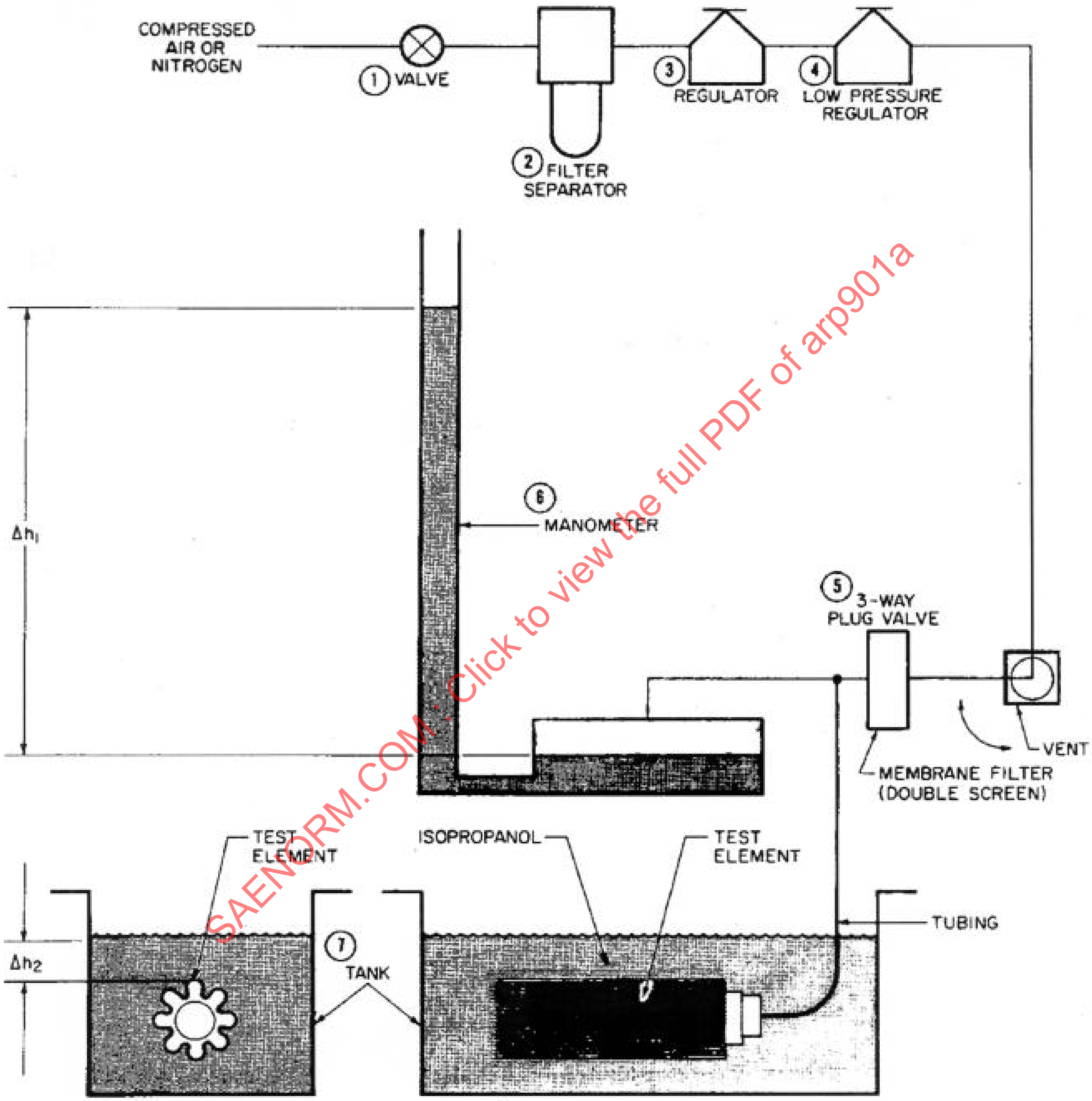

FIGURE 1 - Bubble-Point Test Apparatus

4.1.8 Means for maintaining and measuring test liquid temperature in the range of 79 °F ± 5 °F.

4.2 Test Liquid:

The standard referee test liquid is specified to be American Chemical Society reagent grade isopropanol (isopropyl alcohol) having a surface tension of 21.15 dynes/cm ± 0.10 dynes/cm at 77 °F (25 °C). Substitutions are not permitted. Other test liquids may be used for routine, non-referee bubble-point testing provided that test results must be shown to correlate with, standard referee test results. Typical alternative test liquids include commercial grades of isopropanol, Ethanol 3-190 or other brands of denatured ethanol, and MIL-H-5606 hydraulic oil, JP-5 fuel and other hydrocarbon solvents have also been used but they are generally less suitable, because of larger variations in surface tension and wetting characteristics.

4.3 Test Procedure:

The test element is wetted, attached to the gas source, and tested to locate the approximate area containing the largest pores or holes. The element is turned to bring the bubbling area to the top and additional tests are performed to confirm the largest pore location and to measure the net capillary pressure causing the first bubbles. Bubble-point tests are to be conducted at a measured temperature in the range of 75 °F ± 5 °F.

4.3.1 Inspect the test element to assure that it is clean and dry, free from oil, grease, or other visual contamination or defects which may affect test results.

4.3.2 Immerse the element in the tank to thoroughly wet and saturate the pore structure. Remove and drain excess liquid.

4.3.3 Insert stopper or other sealing device attached to the gas tubing for pressurizing and plug other orifices if necessary.

4.3.4 Immerse the test element just below the liquid surface, gradually increase the gas pressure from zero until the first bubble is observed, and note the location on the element.

NOTE: Air bubbles may be trapped on or within the outer structure resulting in a few spurious bubbles. These bubbles are ignored. The “first bubble” appears in a continuous stream as long as internal gas pressure is maintained. Adequate lighting is necessary for reliable observation. Gas pressure must be increased slowly to allow continuous establishment of equilibrium and prevent overshooting. Mechanical vibration or jarring of the test element must be avoided to prevent upsetting bubble equilibrium which causes erroneous low pressure readings.

4.3.5 Turn the element, if necessary, so that the area emitting the first bubble is nearest the liquid surface.

4.3.6 Release the gas pressure in the filter to allow the pores to be flooded with liquid and repeat the procedure of 3.3.4.

NOTE: Internal gas pressure must be completely released between each bubble determination either by venting to atmosphere or by removing the stopper seal. If complete pore flooding is blocked by gas pressure, spurious low pressure readings may result.

4.3.7 Rotate the test element while maintaining this bubble pressure and note any other areas which may also bubble.

4.3.8 Repeat the procedures of 3.3.4 through 3.3.7 until the area which bubbles at the lowest pressure is located nearest the liquid surface.

4.3.9 Read the lowest pressure on the manometer at which the first bubble is released and record to the nearest 0.1 in of water.

4.3.10 Measure the vertical distance from the point at which the first bubble is emitted to the liquid surface and record to the nearest one-sixteenth of an inch.

4.3.11 Measure the liquid temperature with a thermometer and record to the nearest degree F.

4.4 Calculations:

All bubble-point test results are to be reported as the net standard bubble-point pressure measured in inches of water head. Corrections for depth of immersion and surface tension variation due to temperature may be required. For non-referee tests using liquids other than isopropanol, larger corrections for surface tension are necessary. Standard bubble-point pressure is defined by the following equation.

$$P^{\circ} = \frac{21.15}{S}(P - dh) \qquad \text{(Eq. 1)}$$

P° is standard bubble-point pressure based on isopropanol at 77 °F (25 °C) having a surface tension of 21.15 dynes/cm. Actual liquid surface tension, either directly measured or determined from the temperature measurement of the known liquid, is denoted by S. Measured capillary pressure, P, is corrected by the measured depth of immersion, h, times test liquid density (specific gravity), d. Surface tension and density are shown as a function of liquid temperature for isopropanol and Ethanol 3-190 brand denatured ethanol in Figures 2 and 3 respectively.

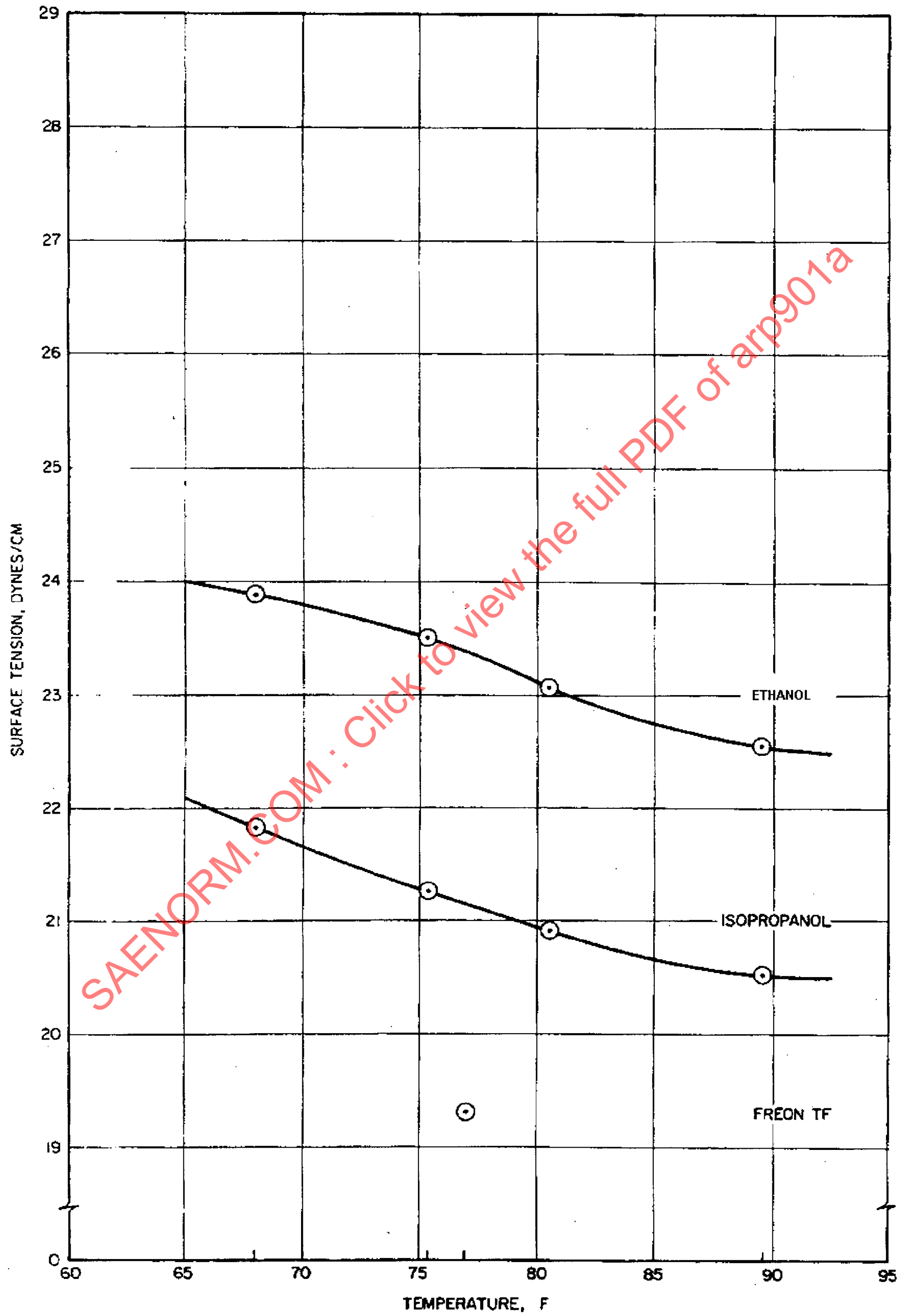

FIGURE 2 - Test Liquid Surface Tension

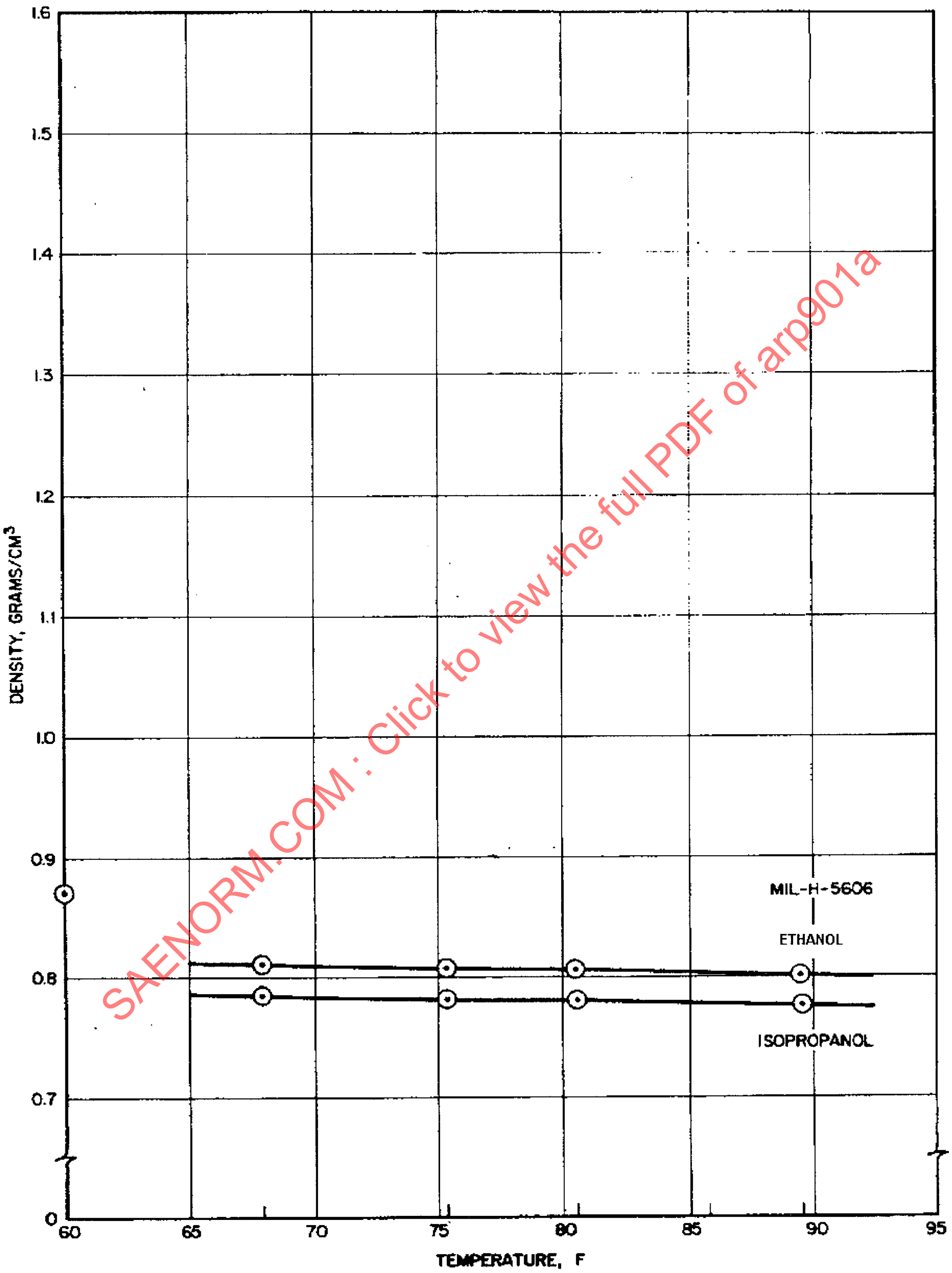

FIGURE 3 - Test Liquid Density

4.4.1 Calculation Examples:

1. A referee bubble-point test using standard isopropanol test liquid is performed on a filter element and the following measurements are determined.

Bubble-point pressure, P	14.6 in WATER COLUMN
Immersion depth, h	0.5 in
Liquid temperature, t.	70 °F

Surface tension and density for isopropanol at 70 °F are found from the curves of Figures 2 and 3.

Surface tension, S	21.65 dynes/cm
Density, d	0.785 gm/cc

This data is substituted in the equation for standard bubble-point pressure:

$$P^\circ = \frac{21.15}{21.65}[14.6 - (0.785)(0.5)] \quad \text{(Eq. 2)}$$

$$= \frac{21.15}{21.65}(14.2)$$

$$= 13.87 = 13.9 \text{ (to nearest 0.1)}$$

The standard bubble-point pressure for this test is determined to be P° = 13.9 in of water based on isoproanol at 77 °F.

2. A routine bubble-point test is conducted according to the referee method on the same filter element described in Example 1, except that Ethanol 3-190 is used as the test liquid. The following measurements are obtained:

Bubble-point pressure, P	16.0 in WATER COLUMN
Immersion depth, h	0.5 in
Liquid temperature, t	70 °F

Surface tension and density for Ethanol 3-190 at 70 °F are read from the curves of Figures 2 and 3.

Surface tension, S	23.80 dynes/cm
Density, d	0.810 gm/cc

4.4.1 (Continued):

This data is used, as in Example 1, in the equation for standard bubble-point pressure.

$$P° = \frac{21.15}{23.80} [16.0 - (0.810 \times 0.5)] \quad \text{(Eq. 3)}$$

$$= \frac{21.15}{23.80} (15.6)$$

$$= 13.88 = 13.9 \text{ (to nearest 0.1)}$$

The standard bubble-point pressure for this test if also found to be P° = 13.9 in of water. The apparent bubble-point difference between Examples 1 and 2 of 1.4 in of water is resolved by proper correction for surface tension difference.

4.4.2 Nomograph Calculations: The numerical calculations specified in 4.4.1 should be used for disputes or other work requiring good accuracy. Routine test calculations may be conveniently solved for surface tension correction only by using the nomograph of Figure 4. The net measured bubble-point pressure (total observed bubble-point less the head due to immersion depth, hxd) is found on the left vertical axis. The appropriate reference mark describing the test liquid and its temperature is located on the diagonal axis. A straight-edge or ruler connecting these two points intersects the right vertical axis at the corresponding standard bubble-point pressure.

Example: A filter element is tested in Ethanol 3-190 at 75 °F and has a bubble-point of 16.8 in with an immersion depth of 1.0 in. Immersion head is approximately 0.8 in (0.81 x 1.0 = 0.8) and net bubble-point Is 16.0 in (16.8 – 0.8 = 16.0). Find 16.0 on the left axis, reading upwards. Find the Ethanol 3-190, 75 °F reference mark on the diagonal axis. Connect both points to intersect the right axis as shown by the dashed line. Read downwards on right axis to find standard bubble-point of 14.3 in at intersection.

5. INSPECTION TEST:

Ordinary bubble-point testing may not require the accuracy nor warrant the cost of the specified referee test. Alternative test liquids such as commercial grade isopropanol, denatured ethanol, or may be used for non-referee tests employing the referee procedure or a functional equivalent modification of the procedure, providing that the user must show proper correlation with the referee test and must report data in terms of “standard bubble-point” as specified for the referee test. For tests which only require assurance that a specified minimum bubble-point is met by a filter element, a simpler “go-no-go” procedure may be used.

5.1 Test Equipment:

Apparatus similar to that specified in 3.1 and shown in Figure 1 is satisfactory. Modifications to meet special auxiliary requirements for cleanliness, higher production rate, etc., are allowable, providing that referee test correlation can be shown for test results.

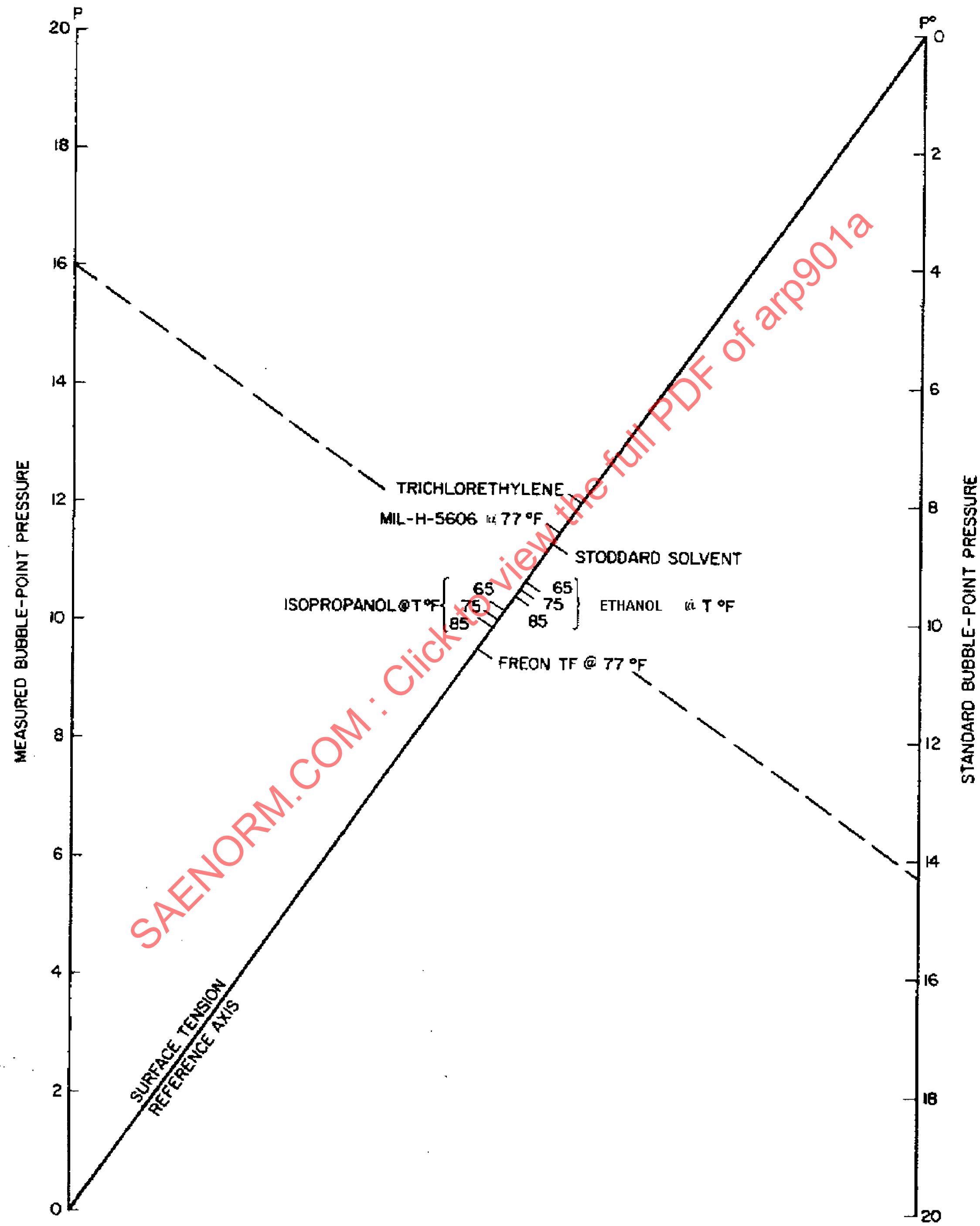

FIGURE 4 - Bubble-Point Correction Nomograph

5.2 Test Liquid:

The standard isopropanol test liquid specified in 4.2 is satisfactory. Alternative test liquids such as commercial isopropanol, Ethanol 3-190 or other brands or denatured ethanol may also be used providing that proper correlation, with referee test results is established after the necessary corrections of 4.4 are applied.

5.3 Calculations:

The go-no-go test is accomplished by applying a specified minimum gross gas pressure and observing the test element for bubble formation. If no bubbles are observed, the element has passed the test. If bubbles are emitted, the element has failed the test and further referee testing or other action may be required. Except for the distinction of passing a minimum bubble-point instead of measuring the actual bubble-point, all of the physical considerations noted in Section 4 for referee tests also apply to inspection tests.

The required minimum gross gas pressure is obtained by use of the equation given in 4.4 and demonstrated in 4.4.1. The specified minimum bubble-point must be expressed as a standard bubble-point or the specific test liquid and temperature must be given. In the latter case, it is convenient to convert the specified bubble-point to the standard bubble-point so as to provide a common basis for calculation and communication. Calculation of the required test pressure is essentially the inverse operation described in 4.4.

Specified bubble-point is multiplied by the ratio of test liquid surface tension/specified liquid surface tension to give the expected minimum net bubble-point. Expected maximum immersion head must be added to this value. This is determined by adding immersion depth to the top of the filter element plus the pleat height (if any) and multiplying this sum by the test liquid specific gravity.

Example: A minimum bubble-point of 16.0 in of water is specified. The specification is found to be based on using Ethanol 3-190 denatured ethanol test liquid at 70 °F with an immersion depth of 0.5 in. The filter element has a pleat depth of 0.280 in. Inspection tests are to be accomplished with commercial grade isopropanol having the same surface tension as standard referee isopropanol at 75 °F. Test fixtures require an immersion depth of 0.25 in/to the top of the filter element. Standard bubble-point is found from Equation 1 and the surface tension and density curves of Figures 2 and 3 for Ethanol 3-190 at 70 °F.

$$P^{\circ} = \frac{21.15}{S}(P\text{-}dh) \qquad \text{(Eq. 4)}$$

Surface tension, Ethanol 3-190 at 70 °F, S	23.80 dynes/cm
Density, Ethanol 3-190 at 70 °F, d	0.810 gm/cc
Immersion depth, h	0.5 in
Total bubble-point, P	16.0 in WATER COLUMN

5.3 (Continued):

$$P^\circ = \frac{21.15}{23.80}[16.0 - (0.810)(0.5)] \quad \text{(Eq. 5)}$$

$$= \frac{21.15}{23.80}(15.6)$$

$$= 13.88 = 13.9 \text{ (to nearest 0.1)}$$

The corresponding standard bubble-point is P° = 13.9 in based on referee isopropanol at 77 °F. The required minimum test pressure is found in a similar manner. The basic equation is rearranged to solve for total capillary pressure, P. Surface tension and density values for isopropanol at 75 °F are determined from Figures 2 and 3. Maximum possible immersion depth is found by adding minimum depth (0.25 in) plus pleat depth (0.280 in) for a total depth of 0.53 in. The calculation is given below:

$$P = \frac{P^\circ S}{21.15} + dh \quad \text{(Eq. 6)}$$

Surface tension, Isopropanol at 75 °F S	21.30 cynes/cm
Density, isopropanol at 75 °F, d	0.780 gm/cc
Immersion depth, maximum total, h	0.53 in
Standard bubble-point, P°	13.9 in WATER COLUMN

$$P = \frac{21.30}{21.15}(13.9) + (0.780)(0.53) \quad \text{(Eq. 7)}$$

$$= 14.0 + 0.413$$

$$= 14.413 = 14.4 \text{ (to nearest 0.1)}$$

Total required test pressure for this example is shown to be 14.4 in water.

5.4 Test Procedure:

After the required total test pressure is established according to the methods of 5.3, the filter elements may be tested for minimum conformance.

5.4.1 Inspect the test element to assure that it is clean and dry, free from oil, grease, or other gross contamination or defects which may affect test results.

5.4.2 Immerse the element in the tank to thoroughly wet and saturate the pore structure. Remove and drain excess liquid.

5.4.3 Insert stopper or other sealing device attached to the gas tubing for pressurizing and plug other orifices if necessary.

5.4.4 Immerse the test element to the predetermined depth, and gradually increase the gas pressure from zero to the calculated test pressure which is required.

NOTE: If the filter element pleats are very deep, it may be necessary to observe the exact point from which the first bubble, if any, is emitted to allow special correction according to the method of Section 4. Other notes of Section 4 regarding recognition of the “first bubble,” adequate lighting, slow rate of pressure increase, and avoiding jarring or vibration also apply to this test.

5.4.5 Maintain the required test pressure and rotate the element at least once so that every part of its circumference is observed.

5.4.6 If no bubbles are observed the element has passed the test. If bubbles are observed the correction specified by the note of 4.4.4 may be applied. If this correction is insufficient to prevent bubbling, the procedure of Section 3 should be used to actually determine the element's standard bubble-point.

6. INTERPRETATION:

All bubble-point test data must be reduced to “standard bubble-point” based on referee isopropanol at 77 °F (25 °C) having a surface tension of 21.15 dynes/cm ± 0.10 dynes/cm according to the method of Section 3. This common basis is necessary for rational data comparison and communication of specification requirements. Standard bubble-point data is used directly for specification purposes without conversion to corresponding “largest pore size” or “absolute rating.” This requirement is necessary because the empirical bubble-point constant used for such calculations is strongly dependent on the type of filter medium being tested. Two filter elements having identical absolute ratings as determined by functional tests may have different standard bubble-points. Bubble-point requirements should be separately determined and specified for each different type of filter element.

Bubble-point data, specifications and acceptance criteria must realistically conform to test method precision, accuracy, and reproducibility. Reported data or specification requirements should be rounded off to include significant digits only, usually to the nearest 0.1 in/of water pressure for bubble-points. Decisions to accept or reject filter elements should consider a reasonable margin for error inherent in the test method.

6.1 Referee Test Interpretation:

Referee tests are usually reproducible within ±0.1 in and accurate within ±0.2 in water pressure. There may be an error of 0.1 in due to rounding off calculated values. Therefore, differences of 0.1 in between separate tests on the same filter element should be resolved in favor of the higher bubble-point figure. If multiple tests of the same element differ by more than 0.5 in, the data should not be averaged, but should be discarded and the cause of this error should be determined before new tests are undertaken.

6.2 Inspection Test Interpretation:

Inspection tests are subject to the same errors as referee tests. Additional error due to bubble-point location along the pleat height may be encountered unless it is corrected according to the note of 4.4.4. As a general rule, filter manufacturers should reject elements having a bubble point below the specified value and filter buyers should accept elements having a bubble-point not more than 0.2 in below the specified value.

7. BUBBLE-POINT PHYSICS:

All bubble-point or capillary pressure methods of pore size measurement depend one same physical phenomena as their basis. A wetting phase is displaced from the pore structure by a non-wetting phase and the pressure required for displacement is measured. Characteristic pore dimensions are rationalized by equating the measured net capillary pressure with the surface tension force opposing the capillary pressure.

7.1 Circular Capillary Model:

The physical basis for bubble-point testing may be rationalized by considering the simple circular capillary model depicted schematically in Figure 5. A moment before the bubble is detached from the pore perimeter shown in cross section, the vector forces F_G and F_L may be considered to be in equilibrium. The total force F_G is due to the capillary gas pressure exerted over the projected area of the bubble or pore. The total force F_L is due to the surface tension of the wetting liquid distributed around the pore perimeter. These vector forces may be equated at equilibrium:

$$F_G = F_L \quad \text{(Eq. 8)}$$

Each of these forces may be analyzed for a circular pore.

$$F_G = PA = P\pi\frac{D^2}{4} \quad \text{(Eq. 9)}$$

where:

P is the net capillary gas pressure (not the measured gross pressure) and
A is the pore area which can be expressed in terms of the pore diameter D for the circular capillary.

$$F_L = S\cos\theta L = S\cos\theta\pi D \quad \text{(Eq. 10)}$$

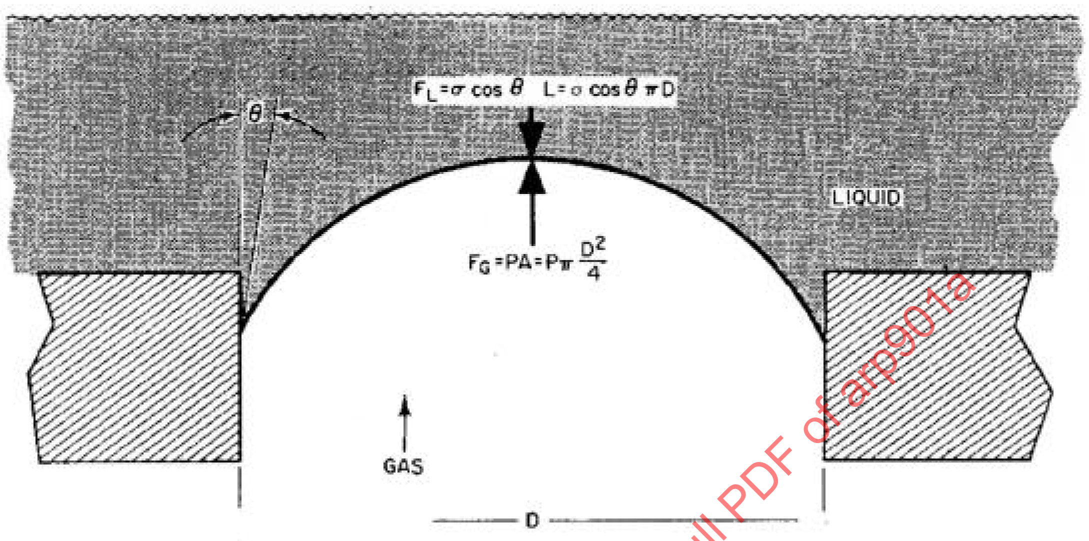

FIGURE 5 - Circular Capillary Model

7.1 (Continued):

where:

S is the surface tension of the wetting liquid in contact with the gas and
θ is the advancing contact angle of the liquid with respect to the pore wall.

The pore perimeter L can also be expressed in terms of the pore diameter D.

The vector force equation may now be expressed in terms of its measurable components.

$$P\pi\frac{D^2}{4} = S\cos\theta\pi D \qquad \text{(Eq. 11)}$$

This equation may be solved for the circular pore diameter.

$$D = \frac{4S\cos\theta}{P} \qquad \text{(Eq. 12)}$$

7.1 (Continued):

The surface tension S and advancing contact angle θ may be measured for a given system while the gas pressure P is determined by the bubble-point test for the largest pore. For example, the surface tension of isopropanol against air is about 21.3 dynes/cm at 75 °F. The contact angle θ may be assumed to be zero if perfect wetting of the pore structure has occurred, although some small positive value of θ may be more likely in physical reality. These constants may be evaluated in consistent dimensional units to provide an equation with a lumped constant where D is expressed in microns and P is in inches of water for convenience:

$$D = \frac{16.06\ S}{P} = \frac{342}{P} \qquad \text{(Eq. 13)}$$

It should be noted that this equation applies directly to the hypothetical circular capillary model only, and cannot be used for other structures which require different assumptions except to define a "circular capillary equivalent" with "perfect wetting."

7.2 General Analysis:

Very few real porous materials approach the structure of the circular capillary model. While the same physical phenomena occur and the same approach to rationalization may be employed for other structure models, a more generalized analysis is required. A pressure differential exists across the curved interface between a gas and liquid phase which can be expressed at any point as a function of the surface tension and bubble curvature.

7.2.1

$$P = S\ (1/R_1 + 1/R_2) \qquad \text{(Eq. 14)}$$

where:

R_1 and R_2 are the principal radii of curvature which define the mean curvature at this point and S is the interfacial tension in the liquid equivalent to the surface tension for air-liquid interfaces.

The shape of a curved interface or meniscus in a capillary is a function of the cross-section and the contact angle of wetting θ. For a circular capillary of diameter D, $R_1 = R_2 = D/2\ (\cos\theta)^{-1}$ and the general equation may be expressed as:

6.2.2 $$P = S\ \frac{2\cos\theta}{D} + \frac{2\cos\theta}{D} = \frac{4S\cos\theta}{D} \qquad \text{(Eq. 15)}$$