AEROSPACE STANDARD

AS598™	REV. A
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(R) Aerospace Microscopic Sizing and Counting of Particulate Contamination for Fluid Power Systems

RATIONALE

This document has been revised to include additional calculation examples along with new and revised figures to clarify the instructions. Some editorial improvements have also been included.

AS598A has been reaffirmed to comply with the SAE Five-Year Review policy.

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1. SCOPE

This SAE Aerospace Standard (AS) defines the materials, apparatus and procedure for microscopic sizing and counting of particulate contamination of fluid power systems by membrane filtration using microscopic counting.

2. REFERENCES

2.1 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.

2.1.1 SAE Publications

Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, Tel: 877-606-7323 (inside USA and Canada) or +1 724-776-4970 (outside USA), www.sae.org.

ARP4285 Aerospace - Evaluation of Particulate Contamination in Hydraulic Fluid - Membrane Procedure

AS1241 Fire Resistant Phosphate Ester Hydraulic Fluid for Aircraft

AS4059 Aerospace Fluid Power - Contamination Classification for Hydraulic Fluids

2.1.2 US Government Publications

Available from the Document Automation and Production Service (DAPS), Building 4D, 700 Robbins Avenue, Philadelphia, PA 19111-5094, Tel: 215-697-9495, https://assist.daps.dla.mil/quicksearch/.

MIL-PRF-83282 Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Aircraft, Metric, NATO Code Number H-537

MIL-PRF-87257 Hydraulic Fluid, Fire Resistant; Low Temperature, Synthetic Hydrocarbon Base, Aircraft and Missile

MIL-PRF-5606 Hydraulic Fluid, Petroleum Base, Aircraft, Missile and Ordnance (Inactive for new design)

2.2 Definitions

CRITICAL CLEANLINESS APPLICATION: An application in which the background contamination encountered during the cleanliness evaluation process would significantly affect the measured cleanliness level.

CALIBRATION FACTOR: A multiplier used to determine the statisitcal quantity of all particulate contaminant on an analysis membrane and is defined as the effective filter area of the analysis membrane divided by the area used in the evaluation of particle counts.

3. PRINCIPLE OF METHOD

A known volume of hydraulic fluid is filtered through a gridded membrane filter to give an even distribution of particulate matter on the membrane. The residual contamination is then sized into five defined size ranges and counted by microscopic analysis.

4. MATERIALS

The following materials shall be used:

1. Membrane filter, 47.00 mm (1.850 inches) in diameter with a pore size less than 1.0 µm. The filter shall have imprinted grid of 3.10 mm ± 0.02 mm (0.122 inches ± 0.001 inches) centers. The color shall be chosen for maximum contrast with the particulate contamination to be observed. As defined in ARP4285, when testing petroleum base hydraulic fluid (MIL-PRF-5606) or synthetic hydrocarbon fluid (MIL-PRF-83282 or MIL-PRF-87257), a white cellulose ester membrane is required. Phosphate Ester fluid, (see AS1241) requires the use of polytetrafluoroethylene (PTFE) type membranes material (or other equivalent material).

2. Membrane filter 25.00 mm (0.984 inches) as filtering disc for rinse dispenser 1 µm or smaller.

3. Plastic film, 0.050 mm (0.002 inches) minimum thickness. The film shall be compatible with sample and rinse liquids (pressure dispenser) or 3 µm (manual dispenser).

4. Petri-dish or Membrane Holder with lid capable of retaining the analysis membrane for microscopic evaluation.

5. APPARATUS

5.1 Filtration Apparatus

Refer to Figure 1.

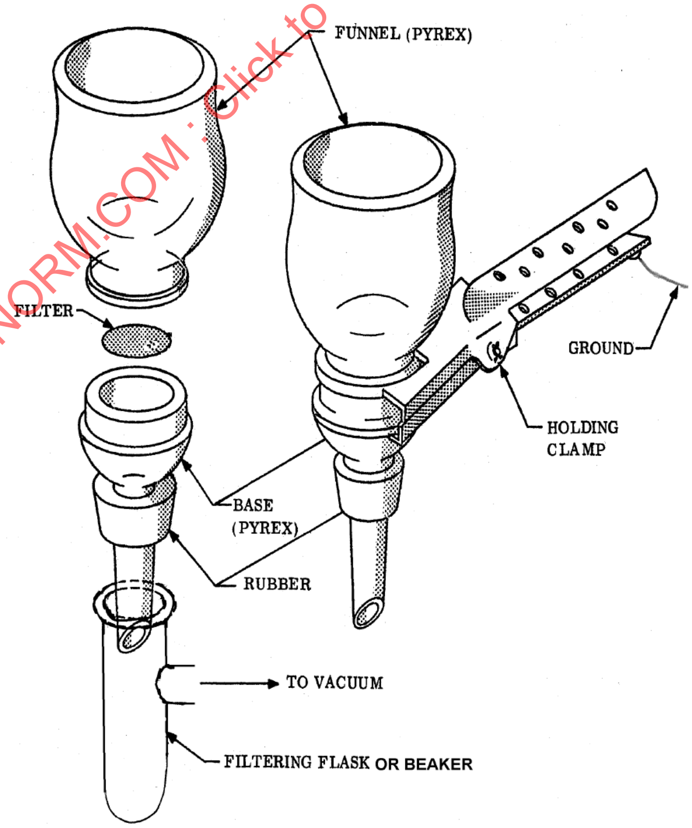

Figure 1 - Diagram of Membrane Filtration Setup

The following fitration apparatus shall be used:

1. Petri dishes - These shall be plastic or glass. If plastic, it shall be compatible with sample and rinse liquids.

2. Sample bottles - These shall be made from glass, be small mouth, with a screw cap, permanently marked to indicate sample volume. The bottle shall have a nominal capacity of 250 mL (8.45 ounces) and a minimum capacity of 150 mL (5.07 ounces) and shall be sealed by means of caps (of phenolic resin), which will not cause contamination. Alternatively, a non-flaking plastic film compatible with the fluid may be used.

3. Filtration funnel - These shall have a lower hole diameter of approximately 35.00 mm (1.378 inches) inner diameter. The effective filtering area shall be calibrated by filtering a contrasting particulate pigment through a membrane filter. The diameter of the residual pigment shall be measured at quadrature diameters with resulting area 960 mm^2 ± 25 mm^2 (1.49 in^2 ± 0.04 in^2). If the receiving funnel is to be used for measuring the sample volume, the funnel shall be calibrated within ±2% of the required volume.

4. Membrane filter support base - These shall be made of either a fritted glass, sintered metal or fine stainless-steel screen may be used. The support shall be so designed as to enable attachment to a vacuum source.

5. Vacuum flask - These shall be appropriately sized to mount the membrane filter support and funnel assembly. Vacuum is applied to the flask to draw the sample fluid through the filter.

6. Holding device - These shall clamp the membrane holder support base and filtration funnel, with a provision to dissipate static electricity.

7. Funnel cover - These shall serve to keep contaminant out of the funnel both before and during use.

8. Vacuum source - These shall have a minimum vacuum of 457 mmHg (18.0 inches of Hg).

9. Forceps - These shall be of flat non-serrated tips.

10. Rinse dispenser - These pressurized containers shall be equipped to pass rinse liquid through a membrane filter with a pore size of 1 µm or finer.

5.2 Particle Count Apparatus

The following particle count apparatus shall be used:

1. Microscope - Binocular or monocular microscopes shall be used.

   NOTE: Stereo microscopes shall not be employed with this procedure.

2. Objectives and oculars (eyepieces) - These shall be in combinations to give magnifications of 50x ± 10x and 100x ± 10x. The higher power objective shall have a minimum Numerical Aperture of 0.15. The ocular shall not be greater than 15x.

3. Ocular Micrometer or Graticule - A linear scale located in the ocular of the microscope shall be used for measurement. The smallest division shall not subtend a distance larger than the smallest particle to be counted at a particular magnification.

4. Mechanical Stage - This shall be capable of traversing the entire area of the membrane filter. It shall have provisions for holding a membrane container.

5. Stage Micrometer - This shall have divisions of 0.1 mm and 0.01 mm (0.004 to 0.0004 inches).

6. Microscope Light - External, Focusing - It shall be equipped with an external adjustable arm to give oblique incident light (15 to 45 degrees from horizontal). It shall provide an illumination of 54 to 65 kilo lumens per square meter (5000 to 6000 ft-c) at the counting surface.

6. REAGENTS

All reagents shall meet local, federal and international regulations for usage and disposal.

6.1 Aqueous

This shall be a liquid detergent solution that leaves no solid residue.

6.2 Alcohols

This shall be ethanol or isopropyl alcohol, acetone free.

6.3 Rinse Solvent

Petroleum Naphta, heptane, hexane, or iso-octane shall be used.

CAUTION: EXTREMELY FLAMMABLE. DO NOT USE NEAR ANY IGNITION SOURCE.

Rinse liquids shall be compatible with the test fluid and shall not leave a residue on drying.

6.4 Cleanliness

The filtration of reagents shall be performed with the apparatus described in 5.1.10. The term "filtered" shall herein refer to 1 µm or finer filtered reagent.

7. PREPARATION OF APPARATUS

7.1 General

The following procedure shall be used:

1. The apparatus shall be thoroughly washed in a solution of liquid detergent and hot water.

2. Rinse with distilled or de-mineralized water.

3. Rinse with filtered alcohol to remove water.

4. Rinse with filtered solvent.

7.2 Sample Bottle Preparation

Follow procedure defined in 7.1, and then allow the bottles to drip dry with open end facing down. Place a piece of plastic film, which has been rinsed with filtered liquid, over the mouth of the bottle. Hold the film while, screwing on the cap to prevent the film from rotating.

8. LIQUID SAMPLES

8.1 Sample Volume

The standard sample should be 100 mL ± 1 mL (3.38 ounces ± 0.03 ounces) except in the following cases: When the particle count from this volume is greater than 100000 or less than 500 particles total, the sample volume may be altered. For counts less than 500 particles, the volume should be a minimum of 200 mL (6.76 ounces). For counts greater than 100000 particles, the volume may be decreased to allow proper particle differentiation.

Note: In all cases, the sample volume shall be recorded.

8.2 Samples Representatively

Samples shall be as representative of the system as possible. Procedures for sampling shall be established by the user. To assure reproducibility, the sampling program should be checked by testing replicate samples from the sample port. Where samples are required from remote facilities two samples shall be supplied, taken concurrently in the same operation.

Note: Fluid samples which contain contaminant that has settled shall be mixed by hand and in an ultrasonic bath.

9. FILTRATION PROCEDURE

9.1 Blank Analysis Filtration

This is the procedure to be completed prior to the sample filtration for critical cleanliness applications.

1. Remove a membrane filter using forceps from the membrane container and rinse with filtered rinse liquid.
2. Place the filter on the membrane filter support, lower the funnel, and secure with the holding device. Cover the funnel.
3. Place equivalent to the volume to be tested of filtered rinse liquid into a sample bottle and agitate.
4. Remove the cover and pour the contents of the bottle into the funnel.
5. Turn on the vacuum and allow the sample to filter until approximately 50.0 mL (1.69 ounces) remain.
6. Pour an additional 50.0 mL (1.69 ounces) of filtered rinse liquid into the bottle and agitate.
7. Pour the contents of the bottle into the funnel, rinsing the funnel walls, and replace the funnel cover. When the fluid filtration rate is excessive, causing a vortex, the vacuum shall be released to allow adequate rinsing of the funnel walls and to eliminate the possibility of upsetting the particle distribution by the rinse liquid.
8. Maintain vacuum on the membrane sample until dry.
9. Remove the cover, holding device and immediately turn off the vacuum.
10. Remove the membrane filter using the forceps and place in a petri dish and label.
11. Perform a particle count as specified in Section 11.
12. If the blank count exceeds 10% of the sample count, the apparatus shall be re-cleaned and the procedure rerun.

9.2 Sample Filtration Procedure

1. Remove a membrane filter using forceps from the membrane container and rinse with filtered rinse liquid.
2. Place the filter on the membrane filter support, lower the funnel, and secure with the holding device. Cover the funnel.
3. Thoroughly agitate the sample and then remove the cap.
4. Remove the funnel cover and pour the sample bottle contents into the funnel.
5. Pour approximately 50.0 mL (1.69 ounces) of filtered rinse liquid in the sample bottle and agitate.
6. Pour the filtered rinsed liquid into the funnel and cover.
7. Turn on the vacuum and allow the sample to filter until approximately 50.0 mL (1.69 ounces) remain.
8. Lift the cover and carefully wash down the funnel walls with rinse liquid. When the rinse liquid rate is excessive, there is a possibility of upsetting the particle distribution (refer to ARP4285).

9. Replace the cover and filter until the membrane is dry.

10. Remove the cover, clamp, and funnel, and then release the vacuum immediately.

11. Using the forceps, transfer the membrane filter to the petri dish or equivalent.

12. Label the petri dish giving the sample volume and identification.

13. Perform a particle count as specified in Section 11.

10. MICROSCOPE CALIBRATION

1. Place the stage micrometer on the mechanical stage and adjust the light.

2. Place the required objective and oculars in the microscope and focus on the micrometer.

3. Calibrate the ocular micrometer, as detailed in Appendix A, located in one eyepiece at each magnification to be used. Do not place the eyepiece containing the ocular micrometer in an adjustable draw tube eyepiece because the calibration will change as the ocular is adjusted. Each operator shall perform this calibration when using the latter microscope. When a binocular is used, the focal length and calibration will change when the interpupillary distance changes. The calibration method requires that the length of the entire linear scale be measured rather than only a portion.

4. The operator shall calculate the number of linear divisions required to measure each range at all magnifications. For example: If an ocular micrometer with 100 divisions measures 250 μm at 50x, then each division would equal 2.5 μm. By calculating the ranges as specified in AS4059, measure as follows: over 100 μm equals 40 division, 50 to 100 μm equals 20 to 40 divisions, 25 to 50 μm equals 10 to 20 divisions.

Figure 2 shows two possible unit areas within a grid square for statistical counting.

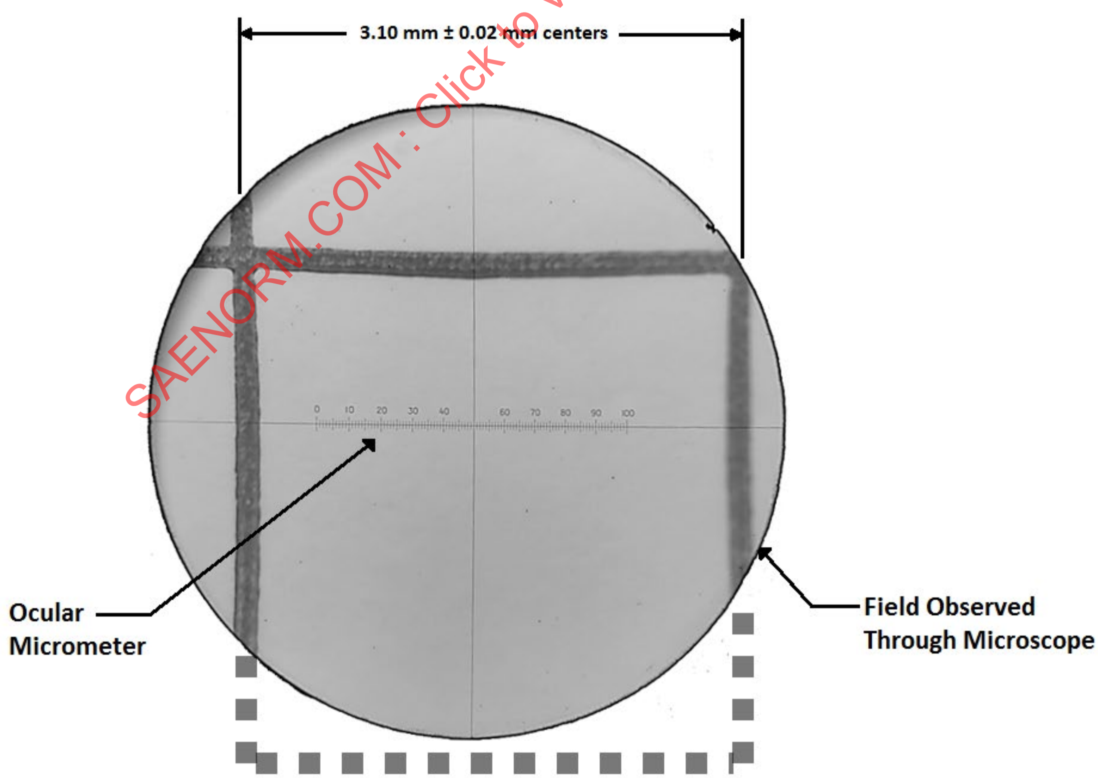

Figure 2 - View of grid square, unit areas and microscope field

The calibration factor may be calculated from the following formula:

$$F = \frac{A_e}{H \times L \times N} \quad \text{(Eq. 1)}$$

where:

F = Calibration factor

N = Number of basic unit areas counted

H = Side of a grid square used in defining the unit area – [normally 3.10 mm ± 0.02 mm (0.122 inches ± 0.001 inches)]

L = Calibrated length of the portion of scale used in defining unit area or ocular micrometer scale - millimeters

A_e = Effective area of membrane used for particle counting [normally 960 mm^2 (1.49 in^2)].

Example:

Using a 10x objective and a 5 mm (0.197 inches) ocular micrometer, counting 10 basic unit areas, the calibration factor would be:

$$L = \frac{5mm}{10x} = 0.5mm$$

$$F = \frac{960mm^2}{3.08mm \times 0.5mm \times 10} = 62.4$$

and the total number of particles of a size range would be:

$$N_t = 62.4 N_c$$

where:

N_c = Number of particles actually counted in the 10 basic unit areas

11. PARTICLE COUNTING PROCEDURE

11.1 General

While certain details of the counting procedure depend somewhat upon specific equipment used, the procedure specified herein shall be followed exactly as stated to provide the required accuracy and reproducibility.

1. The particle size shall be determined by measuring the largest linear dimension.

2. The size ranges shall be as follows: 5 to 15 µm, 15 to 25 µm, 25 to 50 µm, 50 to 100 µm, over 100 µm, and fibers (particles greater than 100 µm which have length to width ratio greater than 10).

3. Remove the container lid and place the sample on the mechanical stage.

4. Select the proper magnification and focus on the membrane grid lines (see Figure 3).

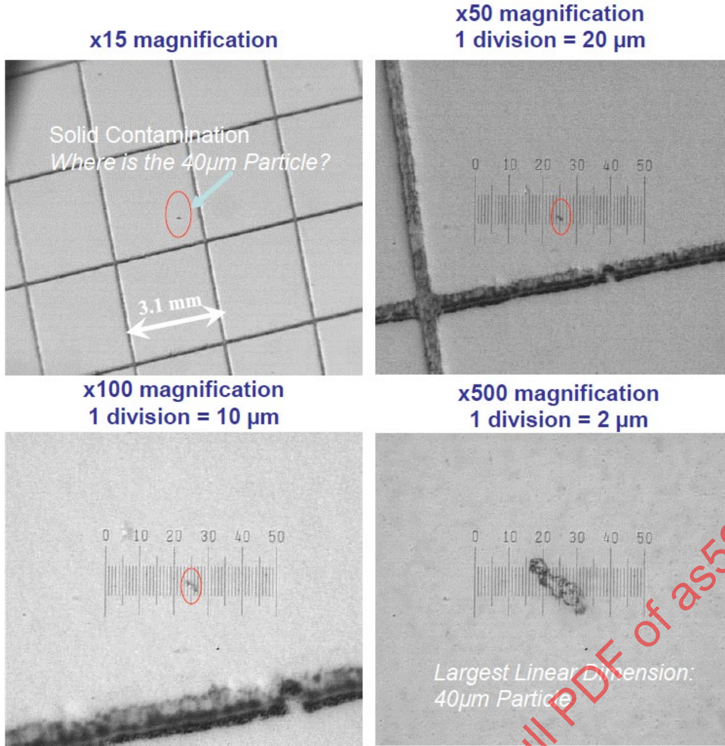

Figure 3 - Magnification selection and particle sizing

5. Turn the petri dish or membrane until the grid lines are aligned with the vertical and horizontal stage axis.

6. Focus the light and then adjust the angle (15 to 45 degrees from horizontal) and intensity to obtain the maximum particle definition.

7. Examine the membrane by scanning the surface to determine that the particles have a random distribution. If the membrane shows evidence of spotty distribution or rings of heavier particle concentration around the outside edge of the filtration area, the statistical counting procedure shall not be used. The liquid sample shall either be re-evaluated, or a total particle count performed.

11.2 Procedure for Sizing and Counting Particles 5 µm and Greater

The following procedure defines a method of sizing and counting particles 5 µm and greater.

1. In obtaining the number of particles of a given particle size range, the number of particles on a representative number of grid squares on the filter membrane is counted. From this count, the total number of particles, which would be present statistically on the total effective filtration area of 100 imprinted grid squares, is calculated.

2. If the total number of particles of a given particle size range is estimated to be between 1 and 50, count the number of particles over the entire effective filtering area.

3. If the total number of particles of a given particle size range is estimated to be between 50 and 1000, count the number of particles in 20 randomly chosen grid squares and multiply this number by 5 to obtain the total statistical particle count.

4. If the total number of particles of a given particle size range is estimated to be between 1000 and 5000, count the number of particles on 10 randomly chosen grid squares and multiply this number by 10 to obtain the total statistical particle count.

5. If the estimated total number of particles of a given size range is estimated to exceed 5000, count the particles within at least 10 randomly chosen unit areas. To arrive at the total statistical count, the sum of the particles counted in 10 or more unit areas is multiplied by the calibration factor.

6. Count the number of particles in each field for each size range by “Gating” the membrane filtration area. As the particles pass by the ocular micrometer measure and record the number of particles in each size range. Gating is the technique where one starts at a reference point and traverses the entire filtration area in a systematic manner. More than one size range can be counted simultaneously providing the magnification is the same.

7. Select a field size so that there are no more than about 50 particles of the size to be counted in the field. Optional fields are: a grid square; a rectangle defined by the width of a grid square and the calibrated length of the ocular micrometer scale; a rectangle defined by the width of the grid square and a portion of the length of the ocular micrometer scale, as shown in Figure 2.

8. For particles improperly oriented relative to the ocular micrometer, an estimate shall be made. The eyepiece containing the ocular micrometer shall not be rotated to size specific particles.

9. If a particle lies on the upper or left boundary line of a counting area, count this particle as if it were within the boundaries of the counting area. Particles on the lower and right-hand boundary lines of the counting area shall not be counted.

12. PARTICLE COUNT CALCULATION AND RESULTS REPORTING

The total particle count for each range shall be calculated using the following formula:

$$\text{Total count} = \frac{A}{F_n \times F_a} \times Pt \qquad \text{(Eq. 2)}$$

where:

A = Filtration area of membrane (normally 960 mm^2 or 1.49 in^2)

F_n = Number of fields (unit areas) counted

F_a = Area of each field (unit area) mm^2 or square inches

Pt = Number of particles in Fn fields or unit areas

Particle counts shall be expressed in particles per 100 mL (3.38 ounces). See 8.1.

The results for each particle size range may be reported as shown in Appendix B.

13. COUNTING PROFICIENCY

13.1 Check Samples

Membrane filters containing contamination, representative of the system to be counted, shall be permanently mounted between glass. These samples should be counted by all operators to determine the mean particle count. This sample may then be sent to outside laboratories for additional counting. Care shall be taken with these slides because the particle count may change as a result of handling.

13.2 Competence

Individual laboratories, with experienced counters, should be able to count with a 10% mean deviation thus providing operator and optical accuracies.

14. DISPUTE CLAUSE

In case of dispute, this clause shall be used to test laboratory competence in filtering and counting particles in clean fluids.

1. Place 200.0 mL (6.763 ounces) of 0.45 µm membrane filtered fluid in a glass bottle cleaned as specified herein.

2. Perform a filtration as specified in Section 9.

3. Count all particles 5 µm and above and record as specified in Section 11.

4. The total particle count shall not exceed a total of 250, including a maximum of 10 particles above 50 µm.

15. NOTES

15.1 Revision Indicator

A change bar (l) located in the left margin is for the convenience of the user in locating areas where technical revisions, not editorial changes, have been made to the previous issue of this document. An (R) symbol to the left of the document title indicates a complete revision of the document, including technical revisions. Change bars and (R) are not used in original publications, nor in documents that contain editorial changes only.

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COMMITTEE A-6, AEROSPACE ACTUATION, CONTROL AND FLUID POWER SYSTEMS