AEROVOX CORPORATION
NEW BEDFORD DIVISION
PLANT OPERATION CHANGE

P.O.C.# 64-1616

EFFECTIVE DATE: 16 Nov 64

AFFECTS	
Q.C. Manual	
Mfg. Proc. Inst.	
Process Spec.	
Test Spec.	X

DISTRIBUTION		
1 X	4	7
2	5	8 X
3	6	9

APPROVAL	
Works Manager	[illegible]
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ORIGIN	
Affected Spec. #	TS-66 K&F Date 6/18/58 & 5/30/63
Originator	E. E. Sasseville
Title	Testing of Askarels and other related Materials.

ACTION: CHANGED [X] ADDED [X] ORIGINAL [ ]

This revision completely replaces TS-66 K and TS-66 F and finalizes the sectionalization begun in Revision F.

Also the following sections were revised:

Section 5 New Procedure in accordance with latest ASTM Method.

Section 7 Added Exhibit #1

Section 12 Added Exhibit #1
Revised Exhibit #3
Revised Exhibit #4

RETURN BOTH TS-66 K and TS-66 F TO THIS DEPARTMENT.

MONS 054079

If this is a change or replacement, the specification or pages that have been changed or replaced must be surrendered to the issuer upon receipt of the revised specification or pages.

Form #BQ-112

1.0 Scope

1.1 This specification covers the method of tests for askarels and other related products.

2.0 Sectionalization

2.1 This specification shall be sectionalized as follows:

Section 1	Issue A	(2 pages)	Specific Gravity
Section 2	Issue A	(1 page)	Color
Section 3	Issue A	(5 pages)	Acidity (Neutralization Number)
Section 4	Issue A	(1 page)	Condition
Section 5	Issue B	(2 pages)	Refractive Index
Section 6	Issue A	(3 pages)	Free Chlorides
Section 7	Issue A	(3 pages)	Pour Point
Section 8	Issue A	(2 pages)	Corrosion Stability
Section 9	Issue A	(1 page)	Resistivity
Section 10	Issue A	(1 page)	Dielectric Constant
Section 11	Issue A	(1 page)	Dissipation Factor
Section 12	Issue B	(4 pages)	Thermal Stability
Section 13	Issue B	(3 pages)	Hydrolysis Stability
Appendix	Issue G	(1 page)	Issue Record

CAUTION: Aroclors are irritating to the skin. The vapors of hot materials are irritating to the eyes and upper respiratory system. If accidental contact is made with the eyes, flush immediately with large quantities of water for at least 15 minutes and get immediate medical attention.

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1.0 Scope

1.1 This section covers the method of testing for specific gravities of askarels and other related products.

2.0 Definition

2.1 The specific gravity of an askarel is the ratio of the weights of equal volume of askarel and water.

2.2 For askarels of low viscosity, both weights are determined with the liquid at a temperature of 60°F (15.56°C) and both weights are corrected for the buoyant effect of air. This characteristic is expressed as "Specific Gravity 60/60°F," wherein the first 60°F refers to the temperature of the askarel and the second 60°F refers to the temperature of the water.

2.3 For askarels of high viscosity, which cannot be tested at 60°F, the standard test temperature is 194°F (90°C) and the specific gravity is recorded as 194/60°F.

3.0 Significance

3.1 Low-viscosity askarels are frequently sold on the basis of volume delivered at 60°F. Delivery is often made on the basis of new weight of product in drums, and the specific gravities are often measured at temperatures other than 60°F. The value of specific gravity at 60°F must be known to compute the volume at 60°F of the askarel delivered.

3.2 Specific gravity is significant in determining the quality or suitability of the askarel for a given application only if the askarel is composed of a single chemical compound. If the askarel is a mixture of two or more compounds, the specific gravity may give only an indication of the relative proportions of the ingredients present.

4.0 Apparatus

4.1 Hydrometer - A hydrometer of the conventional floating glass type, calibrated in terms of specific gravity shall be used. The range of the instrument shall be any suitable portion of the interval between 1.0000 and 2.0000, with the scale divided in 0.001 of a unit of specific gravity, and the scale divisions spaced not less than 0.75 mm (0.03 in.) nor more than 1.5 mm (0.06 in.) apart. The scale shall be so marked that it can be easily read, and the accuracy of the calibration of the hydrometer shall be such that the error at any point of the scale shall not exceed 0.0005.

4.2 Hydrometer Cylinder - The receptacle in which the liquid is tested shall be a cylinder made of clear glass. The inside diameter of the cylinder shall be at least 2.54 cm (1.0 in.) greater than the outside diameter of the hydrometer, and the height of the cylinder shall be such that the column of liquid under test exceeds by at least 2.54 cm (1.0 in.) the submerged portion of the hydrometer after a state of equilibrium has been reached.

...on

...meter - An ASTM thermometer of the total immersion type and conforming to the requirements prescribed in the Specifications for ASTM thermometers (ASTM Designation: E 1)[3], shall be used in taking the temperature of the liquid under test.

...erature-Control Apparatus - Suitable apparatus shall be provided for controlling the temperature of the liquid under test to 60 ± 1°F (15.56 ± 0.56°C) or 194 ± 1°F (90 ± 0.56°C). This may consist of a liquid bath, into which the hydrometer cylinder is placed, or a conditioning chamber in which temperature controlled air is the regulating medium.

5.0 Procedure

5.1 Determine the specific gravity of the askarel at a temperature of 60°F (15.56°C) or 194°F (90°C). Pour the sample to be tested into the clean hydrometer cylinder without splashing, so as to avoid, as much as possible, the formation of air bubbles. If air bubbles are formed, remove them after they have collected on the surface by touching them with a piece of clean blotting or filter paper before placing the hydrometer in the sample. Then put the hydrometer and thermometer in the liquid and place the cylinder in the conditioning medium. Keep them there until the required temperature of the sample as indicated by the thermometer, is reached. An occasional gentle stirring will facilitate reaching the desired temperature. If a liquid bath is used, set-up the apparatus in a location as free from air currents as possible. If an air conditioning chamber is used, shut off any fans used for air circulation as soon as the test temperature is reached, so that disturbance from air currents will be minimized.

5.2 When the liquid under test has reached the test temperature, raise the hydrometer slightly and carefully wipe the stem clean. Then lower the hydrometer slowly into the sample to a level of two smallest scale divisions below that at which it will float, and then release it. When the hydrometer has come to rest, floating freely away from the wall of the cylinder, read and record the specific gravity. In the case of transparent liquids, read the hydrometer with the eye at the level of the surface of the liquid and at the point where the bottom of the meniscus of the liquid intersects the hydrometer scale. In the case of nontransparent liquids, read the hydrometer at the highest point on the scale to which the liquid rises. Correct the hydrometer reading by an amount equal to the height to which the liquid rises on the scale above the main surface of the sample.

5.3 Immediately after the specific gravity is determined, read the thermometer and record the temperature of the sample.

6.0 Precision

6.1 Repeatability - Duplicate determinations should agree within 0.001 unit of specific gravity when the same hydrometer and thermometer are used in the tests.

6.2 Reproducibility - Separate laboratories using different hydrometers and thermometers should obtain duplicate results, agreeing within 0.002 unit of specific gravity.

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1.0 Scope

1.1 This section covers the method of testing for color of askarels and other related products.

2.0 Definition

2.1 The color of an askarel is a numerical value based on comparison with a series of numbered color standards with transmitted light under prescribed conditions.

3.0 Significance

3.1 Color is chiefly significant as an indicator of contamination in askarels. Frequently this contamination is the result of solvent action between the askarel and other materials in the apparatus involved. No definite relationship can be established between color and the physical and electrical characteristics of the liquid; for that reason color in itself has limited value as a measure of condition.

4.0 Preparation of Reference Standards

4.1 Dissolve 1.245 g of potassium chloroplatinate (K_2PtCl_6) containing 0.5 g of platinum, and 1 g of crystallized cobaltous chloride ($CoCl_2 \cdot 6H_2O$) containing about 0.25 g of cobalt in water with 100 ml of hydrochloric acid (HCl, sp gr 1.19). Dilute to 1 liter with distilled water. This solution has a color of 500.

4.2 To prepare reference standards having colors of 5, 10, 15, 20, 25, 30, 35, etc., dilute 0.5, 1.0, 1.5 ml, etc., of the solution prepared as described in Paragraph 4.1 with distilled water to 50 ml in 50-ml tall-form Nessler tubes. Protect the reference standards from evaporation when not in use.

4.3 The unit of color is that color produced by 1 mg of platinum per liter.

5.0 Procedure

5.1 Observe the color of the sample by filling a matched Nessler tube to a height equal to that in the reference standard tubes with the liquid to be examined and comparing it with the reference standards. Determine the color by looking vertically downward through the tubes upon a white or mirrored surface placed at such an angle that light is reflected through the columns of liquids.

5.2 This method is good only up to a color intensity of about 100. For reading higher values the sample should be diluted with water-white benzol and the color obtained by multiplying by the dilution ratio.

6.0 Recording colors

6.1 Record colors between 1 and 100 to the nearest 5.

6.2 Record colors between 101 and 300 to the nearest 10.

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7.0 Limit of test

7.1 Record colors above 300 in accordance with the Method of Test for Color of Lubricating Oil and Petrolatum by Means of ASTM Union Colorimeter (ASTM Designation: D 155).

1.0 Scope

1.1 This section covers the methods of testing for the Neutralization Number of Askarels and other related materials.

2.0 Definition

2.1 Total Acid Number - The total acid number is the number of milligrams of potassium hydroxide (KOH) required to react with 1 g of askarel when tested as prescribed.

2.2 Strong Base Number - The strong base number is the number of milligrams of KOH equivalent to the acid required to react with 1 g of askarel when tested as prescribed.

3.0 Significance

3.1 In the inspection of unused askarels, the neutralization value is of importance as a quality index of purity.

3.2 Since askarel is not subject to deterioration by oxidation, small changes in the neutralization value of used askarels may indicate the solution of basic or acidic materials from the various solid materials in contact with the askarel or the deterioration of such soluble materials to form basic or acidic materials. A large change in acidity may indicate decomposition of the askarel by an electric arc.

4.0 Apparatus

4.1 Buret - A 50-ml buret graduated in 0.1-ml subdivisions, or a 10-ml buret graduated in 0.05-ml subdivisions.

5.0 Purity of Reagents and Materials

5.1 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. 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.

5.2 References to water shall be understood to mean distilled water.

6.0 Reagents

6.1 Hydrochloric Acid Solution, Standard Alcoholic (0.1 N). Mix 9 ml of concentrated hydrochloric acid (HCl, sp gr 1.19) with 1000 ml of anhydrous isopropyl alcohol (containing less than 0.9 percent water). Standardize frequently enough to detect normality changes of 0.0005, preferably by electrometric titration of approximately 8 ml (accurately measured) of the 0.1 N alcoholic KOH solution diluted with 125 ml of carbon dioxide-free water.

6.2 Methyl Orange Indicator Solution - Dissolve 0.1 g of methyl orange in 100 ml of water.

p-Naphtholbenzein Indicator Solution - The p-Naphtholbenzein must meet the specifications given in the Appendix. Prepare a solution containing 10 g of p-naphtholbenzein per liter of titration solvent. (Paragraph 6.5).

6.4 Potassium Hydroxide Solution, Standard Alcoholic (0.1 N). Add 6 g of solid KOH to approximately 1 liter of anhydrous isopropyl alcohol (containing less than 0.9 percent water) in a 2-liter Erlenmeyer flask. Boil the mixture gently for 10 to 15 min., stirring to prevent the solids from forming a cake on the bottom. Add at least 2 g of barium hydroxide ($Ba(OH)_2$) and again boil gently for 5 to 10 min. Cool to room temperature, allow to stand for several hours, and filter the supernatant liquid through a fine sintered-glass or porcelain filtering funnel; avoid unnecessary exposure to carbon dioxide (CO_2) during filtration. Store the solution in a chemically resistant dispensing bottle out of contact with cork, rubber, or saponifiable stopcock lubricant and protected by a guard tube containing soda lime or soda asbestos (Ascarite). Standardize frequently enough to detect changes of 0.0005 N, preferably against pure potassium acid phthalate in about 100 ml of CO_2-free water, using phenolphthalein to detect the end point.

6.5 Titration Solvent - Add 500 ml of toluene and 5 ml of water to 495 ml of anhydrous isopropyl alcohol.

7.0 Preparation of Used Oil Samples

7.1 Strict observance of the sampling procedure described in paragraph 7.2 is necessary, since the sediment itself is acidic or basic or has absorbed acidic or basic material from the sample. Failure to obtain a representative sample causes serious errors.

7.2 Heat the sample of used oil to 60 ± 5°C in the original container and agitate until all sediment is homogeneously suspended in the oil. If the original container is of opaque material, or if it is more than three-fourths full, transfer the entire sample to a clear glass bottle having a capacity at least one third greater than the volume of the sample, and transfer all traces of sediment from the original container to the bottle by violent agitation of portions of the sample in the original container. After complete suspension of all sediment, strain the sample or a convenient aliquot through a 100-mesh screen for the removal of large contaminating particles.

8.0 Procedure for Total Acid Number

8.1 Into a 250-ml Erlenmeyer flask, introduce a weighed quantity of the sample as given in Table I. Add 100 ml of the titration solvent and 0.5 ml of the indicator solution, and without stoppering, swirl until the sample is entirely dissolved by the solvent. If the mixture assumes a yellow-orange color, proceed as directed in Paragraph 8.2; if it becomes green or green-black, proceed as directed in Section 9.0.

8.1 (Continued)

TABLE I - SIZE OF SAMPLE

Total Acid Number or Total Base Number	Size of Sample, g	Sensitivity of Weighing, g
NEW OR LIGHT-COLORED OILS		
0.0 to 3.0	20.0 ± 2.0	0.05
3.0 to 25.0	2.0 ± 0.2	0.01
25.0 to 250.0	0.2 ± 0.02	0.001
USED OR DARK-COLORED OILS		
0.0 to 25.0	2.0 ± 0.2	0.01
25.0 to 250.0	0.2 ± 0.02	0.001

8.2 Titrate immediately at a temperature below 30°C. Add 0.1 N KOH solution in increments and swirl to disperse the KOH as necessary. Shake vigorously near the end point, but avoid dissolving carbon dioxide (CO_2) in the solvent. (In the case of acidic oils, the orange color changes to a green or green-brown as the end point is approached). Consider the end point definite if the color change persists for 15 sec. or if it reverses with two drops of 0.1 N HCl.

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8.3 Blank - Make a blank titration on 100 ml of the titration solvent and 0.5 ml of the indicator solution, adding 0.1 N KOH solution in 0.05- or 0.1-ml increments. Record the quantity of 0.1 N KOH solution required to reach the end point (orange to green).

9.0 Procedure for Strong Base Number

9.1 If the titration solvent containing the dissolved sample assumes a green or greenish-brown color after the indicator is added (Section 8.1), carry out the titration as described in Section 8.2, but use 0.1 N HCl and titrate until the green-brown color changes to orange.

9.2 Blank - Make a blank titration as directed in Section 8.3.

10.0 Procedure for Strong Acid Number

10.1 Introduce approximately 25 g of a representative sample, weighed to the nearest 0.1 g, into a 250-ml separatory funnel and add 100 ml of boiling water. Shake vigorously and drain the water phase, after separation, into a 500-ml casserole. Extract the sample twice more with 50-ml portions of boiling water, adding both extracts to the casserole. To the combined extracts add 0.1 ml of methyl orange indicator solution and, if the solution becomes pink or red, titrate with 0.1 N KOH solution until the solution becomes golden brown in color. If the initial color is not pink or red, report the strong acid number as zero.

10.2 Blank - Into a 250 ml Erlenmeyer flask, introduce 200 ml of the same boiling water as used for the sample titration. Add 0.1 ml of methyl orange indicator solution. If the indicator color is yellow-orange, titrate with 0.1 N HCl to the same depth and shade of color obtained in the titration of the sample. If the indicator color is pink or red, titrate with 0.1 N KOH solution to the same end point as that used in the sample titration.

11.0 Calculations

11.1 Calculate the total acid number as follows:

Total acid number, mg of KOH per g

$$= \frac{(A - B) N \times 56.1}{W}$$

where:

A = milliliters of KOH solution required for titration of the sample (Section 8.2)
B = milliliters of KOH solution required for titration of the blank (Section 8.3)
N = normality of the KOH solution, and
W = grams of sample used

11.2 Calculate the strong-acid number as follows:

11.2.1 If the blank titration is made with acid:

Strong-acid number, mg of KOH per g = $\frac{(CN + Dn) \times 56.1}{W}$

where:

C = milliliters of KOH solution required to titrate the water extract (Sec. 10.1)
N = normality of the KOH solution
D = milliliters of HCl solution required to titrate the blank solution (section 10.2).
n = normality of the HCl solution, and
W = grams of sample used.

11.2.2 If the blank titration is made with base:

Strong-acid number, mg of KOH per g

$$= \frac{(C - D) N \times 56.1}{W}$$

where:

C = milliliters of KOH solution required to titrate the water extract (section 10.1).
D = milliliters of KOH solution required to titrate the blank solution (section 10.2).
N = normality of the KOH solution, and
W = grams of sample used

11.3 Calculate the strong base number as follows:

Strong-base number, mg of KOH per g

$$= \frac{(En + FN) \times 56.1}{W}$$

where:

E = milliliters of HCl solution required for titration of the sample (Section 9)
n = normality of the HCl solution
F = milliliters of KOH required for titration of the acid number blank
N = normality of the KOH solution
W = grams of sample used.

1.0 Naphtholbenzein shall conform to the following requirements:

1.1 Appearance: Red amorphous powder

1.2 Chlorides: Less than 0.5 percent

1.3 Solubility: Ten grams shall dissolve completely in 1 liter of titration solvent.

1.3.1 Titration solvent: Add 500 ml of toluene and 5 ml of water to 495 ml of anhydrous isopropyl alcohol. The titration solvent should be made up in large quantities.

1.4 Minimum absorbance: Exactly 0.1000 g of sample is dissolved in 250 ml of methanol. Five milliliters of this solution is made up to 100 ml with pH 12 buffer. This final dilution should have a minimum absorbance of 1.20 when read at the 650 Mm peak using a Beckman DU spectrophotometer, 1 cm cells and water as the blank.

1.5 pH range

1.5.1 Indicator turns to the first clear green at a relative pH of 11 ± 0.5 when tested by the method for pHr range of p-naphtholbenzein indicator as described in paragraph 6.3.

1.5.2 Requires not more than 0.5 ml of 0.01N KOH above that for blank to bring indicator solution to the first clear green.

1.5.3 Requires not more than 1.0 ml of 0.01N KOH above that for blank to bring indicator solution to a blue color.

1.5.4 Initial pHr of indicator solution is at least as high as that of the blank.

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1.0 Scope

1.1 This section covers the testing of askarels and other related materials for condition.

2.0 Condition

2.1 The condition of the liquid under examination is determined by inspection while it is in the original sample container. If container is of amber color, agitate contents and pour out a 1000 ml sample into an analytically clean beaker.

2.2 The condition should be reported as "clear", "cloudy", "Sediment", "sludge", or "water".

2.3 Observation should include any traces of water, either as emulsion or in the free state.

2.4 If the liquid is not clear, the precipitation value shall be run and the solid matter identified as "sludge" or "insoluble sediment" by washing with acetone.

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1.0 Scope

1.1 This section covers the methods of testing for the refractive index of askarels and other related materials.

2.0 Definition

2.1 The refractive index of a substance may be defined as the ratio of the velocity of light in a vacuum to its velocity in the substance, or as the ratio of the sine of the angle of incidence to the sine of the angle of refraction.

3.0 Significance

3.1 The refractive index of an askarel varies with its composition, and with the nature and amount of contaminants held in solution. Where the refractive index of an askarel when new is known, determinations made on the same askarel after periods of service may form a basis for estimating any change in composition or the degree of contamination acquired through solution.

4.0 Apparatus

4.1 Refractometer - An Abbe refractometer with scale graduated directly in terms of the refractive index of the D line of sodium at a temperature of 20°C.

4.2 Water-Temperature Control - A device to regulate the temperature (usually 20°C) of the water supply to the refractometer.

4.3 Sodium Arc Lamp, G.E. #05 CL (Macalaster Scientific Corp., their #21597).

5.0 Sampling

5.1 Obtain the sample of askarel in accordance with the Method for Sampling Askarels (ASTM Designation: D 1809).

6.0 Procedure

6.1 Periodically check the adjustment of the refractometer with the reference standard supplied with the instrument.

6.1.1 Clean the instrument in accordance with the instructions furnished by the manufacturer before each sample is tested. If no special instructions are provided by the maker, clean the prisms with clinical ether and surgical cotton, and wipe the surfaces immediately with dry cotton. Allow two minutes for temperature equalization before applying the sample to the prism.

6.2 Adjust the bath thermostat to 25°C reading this temperature on the refractometer thermometer on the discharge side. The flow of water should be continued long enough before a sample is tested to show that a constant temperature has been reached.

6.3 Apply the sample to the surface of the refracting prism or to the capillary opening with a round-end glass rod, and allow two minutes for temperature equalization.

6.4 Read the refractive index to three decimal places and estimate the fourth place.

6.5 Test additional samples until three readings have been secured that do not show a spread of more than two units in the fourth decimal place. Record the refractive index as the average of these readings rounded off to four decimal places.

6.6 Read the compensator dial in two positions and average the values.

7.0 Report

7.1 The report shall include the following:

7.1.1 Refractive index, reported to the fourth decimal place, and the temperature at which the test was made.

8.0 Accuracy

8.1 The accuracy of the refractive index determination made by this method is three units in the fourth decimal place.

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1.0 Scope

1.1 This section covers the methods of testing for free chlorides in Askarels and other related materials.

2.0 Definition:

2.1 Askarels may contain free chlorides as a result of imperfect refining or of contamination from other sources. This test indicates the presence of free chlorides by precipitation of insoluble salts through suitable analytical procedures, and is reported in parts per million.

3.0 Significance

3.1 Free chlorides in askarels are undesirable because of their possible corrosive action in the presence of moisture. In unused askarels, they may result from imperfect refining or contamination. In askarels in service, they may also result when the material is subjected to an electric arc or heated above a limiting temperature, which depends on the type of askarel.

4.0 Askarels

4.1 Electrometric Titrimeter

4.2 Electrodes: Silver and glass

4.2.1 Although the silver billet-glass electrode system is preferred, any other standard reference electrode may be used provided that there is no chloride contamination of the sample. However, a silver-silver chloride electrode system should not be used.

4.2.2 Beckman Catalog No. 39261 Billet Electrode and Catalog No. 40498 Glass Electrode have been found satisfactory for this purpose.

4.3 Buret - Microburet, 1.0-ml capacity.

4.4 Magnetic Stirrer, with glass-covered or tetrafluoroethylene (Teflon)- coated stirring bar.

5.0 Reagents

5.1 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. 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.

5.2 Purity of Water - Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to the specifications for Reagent Water (ASTM Designation: D 1193). In addition, the water shall be chloride-free, as detected by this test.

5.3 Acetone, containing no titratable chloride.

5.4 Nitric Acid (1:69) - Mix 1 volume of concentrated nitric acid (HNO_3, sp gr 1.42) in 69 volumes of water.

5.5 Silver Nitrate, Standard Solution (0.005 N). - Prepare by dissolving 0.8495 gm of silver nitrate ($AgNO_3$) in 1000 ml of water containing 3 ml of concentrated nitric acid (HNO_3, sp gr 1.42), or by diluting 5 ml of 0.1 N $AgNO_3$ to 100 ml. Dry the $AgNO_3$ overnight in a desiccator before making up the solution. Both the solid material and the solution must be protected from light by storage in brown glassware in the dark.

6.0 Preparation of Apparatus

6.1 In the determination of low levels of chloride content, extreme care must be used in the cleaning and handling of all apparatus. Glassware should receive a final rinse in water and should not be touched with the hands afterwards.

7.0 Procedure

7.1 Weigh 100 g of askarel into a clean 250-ml beaker. Weigh to ± 0.5 g.

7.2 Add 100 ml of acetone measured in a 100-ml graduate. Add 2 ml of HNO_3 (1:69).

7.3 Titrate with 0.005 N $AgNO_3$ solution, using a magnetic stirrer with glass-covered or tetrafluoroethylene (Teflon)- coated stirring bar and an electrode system. Normal samples of askarel require extremely small amounts of $AgNO_3$ solution, and for this reason titrate using 0.01 ml additions and allowing sufficient time for equilibrium to be established before recording the emf change. If a change of less than 1 mv per 0.01 ml addition is observed for three or four 0.01 ml increments, larger additions of $AgNO_3$ solution may be used, for instance 0.05 ml, until such a change is observed. Then reduce the additions to 0.01 ml again to complete the titration. The end point normally is defined by two 50 mv changes. A normal titration would yield data such as are shown in Table I.

TABLE I - TYPICAL DATA FOR TITRATION

mv	Δ mv	ml	Δ ml	Δ mv/Δml X 10-2
400	0	0.06	0	0
392	8	0.07	0.01	8
352	8	0.08	0.01	8
341	11	0.09	0.01	11
321	20	0.10	0.01	20
271	50	0.11	0.01	50
221	50	0.12	0.01	50
201	20	0.13	0.01	20
285	16	0.14	0.01	16

NOTE 2: Caution - Samples should not be exposed nor the test conducted in an atmosphere containing appreciable quantity of chlorides, as contamination will occur and erroneous results will be obtained.

7.4 To calculate the change per 0.01 ml observed, divide the change in milli-volts by the volume of $AgNO_3$ solution. By plotting Δ mv/Δ ml versus milliliters, the end point may be found to the nearest 0.001 ml. This gives a sensitivity of $\pm$ 0.0017 ppm. To define the end point to $\pm$ 0.01 ml no plotting is necessary, and a sensitivity of $\pm$ 0.02 ppm is assumed.

7.5 Repeat Section 7.2 and 7.3, using a blank sample of acetone. The value obtained on the blank, in addition to its use as a correction to the value obtained on the sample, can also serve as an indicator of cleanliness of the glassware, purity of the reagents, and absence of chlorides from the atmosphere. Normally 1 drop of 0.005 N $AgNO_3$ solution should be sufficient to titrate the blank.

8.0 Calculation

8.1 Calculate the chloride content as follows:

Chloride content, ppm = 1.77 (A - B)
where:
A = milliliters of 0.005 N $AgNO_3$ solution used to titrate the sample, and
B = milliliters of 0.005 N $AgNO_3$ solution used to titrate the blank.

9.0 Precision

9.1 Repeatability - Interlaboratory testing indicates that the difference between two measurements in one laboratory should not exceed (95 percent of the time):

Approximate chloride content, ppm	Repeatability, ppm
0.1	0.002
1.0	0.003
4.0	0.004

9.2 Reproducibility - The difference between two laboratories, each reporting the average of two determinations, should not exceed (95 percent of the time):

Approximate chloride content, ppm	Reproducibility, ppm
0.1	0.050
1.0	0.082
4.0	0.189

1.0 Scope

1.1 This section covers the method of testing for the Pour Point of askarels and other related materials.

2.0 Definition

2.1 The pour point of an askarel may be defined as the lowest temperature at which the askarel will pour or flow under the prescribed conditions of test.

3.0 Significance

3.1 Askarels, like oils, tend to solidify if the temperature is lowered sufficiently. The pour point of an askarel gives an indication of the temperature below which it may not be possible to pour or remove the material from its container.

3.2 The pour point of a capacitor askarel is important as an index of the lowest temperature to which the material may be cooled without its circulation in the casing being seriously impeded. The pour point of a capacitor askarel is also of importance in the design of certain types of capacitors for low temperature applications.

4.0 Apparatus

4.1 The following apparatus shown in Exhibit #1 shall be used.

4.1.1 Test jar: A test jar of clear glass, cylindrical form, flat bottom, approximately 1-3/16" to 1-5/16" in inside diameter and 4-1/2" to 5" in height. An ordinary 4 oz. oil sample bottle may be used if it meets these requirements, and no test jar is available.

4.1.2 Thermometer: An ASTM Cloud and Pour Test Thermometer having a range of - 38°C to + 58°C or - 36°F to + 120°F, as specified, and conforming to the requirements for thermometer 5C or 5F, respectively, as prescribed in ASTM Specification E-1, or an ASTM Low Cloud and Pour Thermometer having a range of - 80°C to + 20°C or -112°F to +70°F, as specified, and conforming to the requirements for thermometer 6C or 6F, respectively, as prescribed in ASTM Specification E-1.

4.1.3 Cork: A cork to fit the test jar, bored centrally to take the test thermometer.

4.1.4 Jacket: A jacket of glass or metal, water tight, of cylindrical form, flat bottom, about 4-1/2" in depth, with inside diameter 3/8" to 1/2" greater than the outside diameter of the test jar.

4.1.5 Disk: A disk of cork or felt, 1/4" in thickness and of the same diameter as the inside of the jacket.

4.1.6 Gasket: A ring gasket, about 3/16" in thickness, to fit snugly around the outside of the test jar and loosely inside the jacket. This gasket may be made of cork, felt or suitable material, elastic enough to cling to the test jar and hard enough to hold its shape. The purpose of the ring gasket is to prevent the test jar from touching the jacket.

4.1.7 Cooling Bath: A cooling bath, of a type suitable for obtaining the required temperatures. The size and shape of the bath are optional but a support, suitable for holding the jacket firmly in a vertical positon is essential.

4.1.7.1 For determinations of pour points below 50°F, two or more baths should be at hand. The required bath temperatures may be maintained by refrigeration if available, otherwise by suitable freezing mixtures.

NOTE: The freezing mixtures commonly used are as follows:

	For temperatures down to:
1. Ice and water	50°F
2. Crushed ice and sodium chlorides	10°F
3. Crushed ice and calcium chloride crystals.	-15°F
4. Solid carbon dioxide and acetone (or gasoline) (Per ASTM D-97)	-70°F

5.0 Procedure

5.1 Pour the oil into the test jar to a height of not less than 2 nor more than 2-1/2". Mark the jar to indicate the proper level. When necessary, heat the oil in a water bath just sufficiently for pouring into the test jar.

5.2 Close the test jar tightly by the cork, carrying the test thermometer in a vertical position in the center of the jar with the thermometer bulb immersed so that the beginning of the capillary shall be 1/8" below the surface of the oil.

5.3 Heat the oil, without stirring, to a temperature of 115°F in a bath maintained at not higher than 118°F. Cool the oil to 90°F in air or in a water bath approximately 77°F in temperature.

5.3.1 Heat oils on which a pour point below -30°F is expected as above with the high cloud- and pour- test thermometer in position, cool to 60°F, place the low cloud- and pour- test thermometer in position, and then place the assembly in the jacket.

5.3.2 Heat oils on which a pour point of above 90°F is expected to 115°F or to a temperature 15°F above the expected pour point, with the high cloud- and pour- test thermometer in position, and immediately introduce the test jar into the jacket.

5.4 Place the disk in the bottom of the jacket and insert the test jar, with the ring gasket 1" above the bottom, into the jacket. The disk, gasket and inside of the jacket shall be clean and dry.

5.5 After the oil has cooled enough to allow the formation of crystals, take great care not to disturb the mass of the oil nor to permit the thermometer to shift in the oil. Any disturbance of the spongy network of crystals will lead to low and fictitious results.

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5.6 Maintain the temperature of the cooling bath at 30°F to 35°F. Support the jacket, containing the test jar, firmly in a vertical position in the cooling bath so that no more than 1" of the jacket projects out of the cooling medium.

5.7 Beginning at a temperature 20°F before the expected pour point, at each test thermometer reading that is a multiple of 5°F, remove the test jar from the jacket carefully and tilt it just enough to ascertain whether there is a movement of the oil in the test jar. The complete operation of removal and replacement shall require not more than 3 seconds.

5.7.1 If the oil has not ceased to flow when its temperature has reached 50°F, place the test jar in the jacket in a second bath maintained at a temperature of 0° to +5°F. If the oil has not ceased to flow when its temperature has reached 20°F, place the test jar in a third bath maintained at a temperature of -30° to -25°F. For determinations of very low pour points, additional baths should be maintained with successively lower temperature differentials of about 30°F. In each case transfer the test jar when the temperature of the oil reaches a point 50°F above the temperature of the new bath.

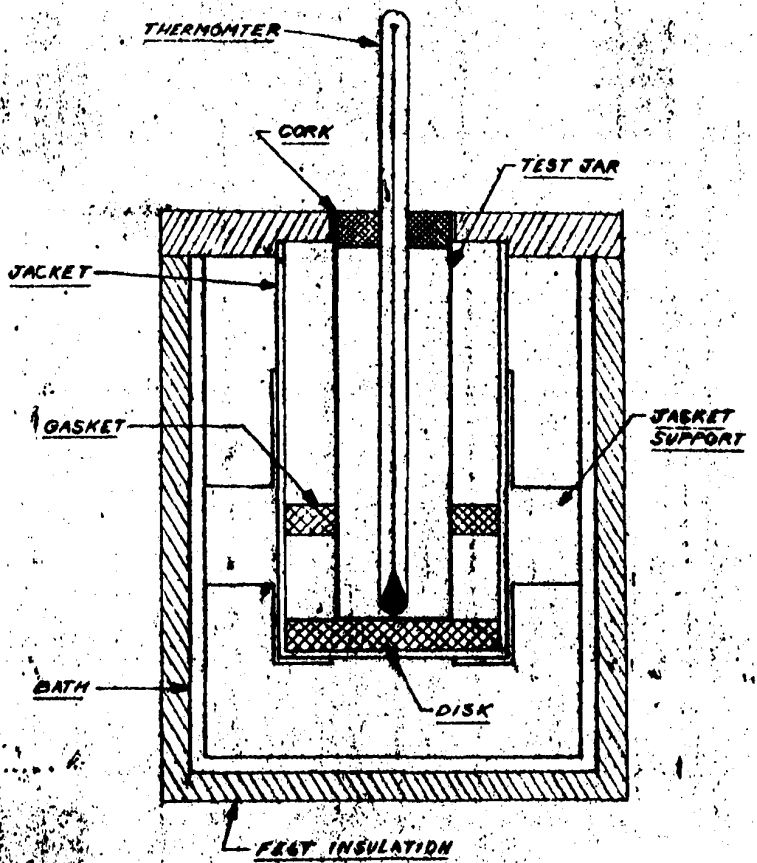
5.8 At no time place the cold test jar directly in the cooling medium. As soon as the oil in the test jar does not flow when the jar is tilted, hold the jar in a horizontal position for exactly 5 seconds, as noted by a stop watch or other accurate timing device, and observe carefully. If the oil shows any movement under these conditions, replace the test jar immediately in the jacket and repeat a test for flow at the next temperature 5°F lower.

5.9 Continue the test in this manner until a point is reached at which the oil in the test jar shows no movement when the test jar is held in a horizontal position for exactly 5 seconds. Certain oils tend to move as a whole and should be very closely observed. Record the reading of the test thermometer at this temperature, corrected for error if necessary. Take the pour point as the temperature 5°F above this solid point.

NOTICE

TOLERANCE UNLESS OTHERWISE SPECIFIED: DECIMALS ± .005 FRACTIONS ± 1/64 ANGLES ± 1/2 DEGREE

DRAWING NO. TS 66 ALT



MONS 054099

AEROVOX
NEW BEDFORD
MASSACHUSETTS, U.S.A.

MATERIAL	SPEC.	FINISH SPEC.	WT. UNIV	FINISH	RAW	LB
DRAWN BY SASSEVILLE	5/15/64			CHECKED BY		DATE
ENG. APPR.	DATE			MFG. APPR.		DATE

TITLE: FOUR TEST APPARATUS

DRAWING NUMBER: SECTION 7 EXHIBIT #1 ALT

A	ORIGINAL				
ALT.	DESCRIPTION	DATE	BY	CK.	ENG. APP.

ALTERATIONS

1.0 Scope

1.1 This section covers the method of testing for corrosion stability of askarels and other related materials.

2.0 Apparatus

2.1 300 ML Erlenmeyer flask (Ground Glass type)

2.2 Water cooled condenser (Ground Glass type to fit flask)

NOTE: Condenser and flask should be painted black on the exterior so as to preclude the effects of light since the material being tested is photo sensitive.

2.3 Accurately controlled bath or electric heating mantle capable of maintaining a temperature of 210°C ± 5°C.

2.4 Scale: analytical

3.0 Material

3.1 200 ml of oil being tested

3.2 One gram of accurately weighed aluminum foil. (99.8% purity and approximately 3 mils thick).

3.2.1 This aluminum foil should be of the type known in the foil industry as "Dry Foil".

4.0 Reagents (all reagents to be chemically pure, c.p.)

4.1 Deionized water

4.2 Acetone

4.3 Benzene

4.4 Chloride free ether

5.0 Procedure

5.1 Glass apparatus (glass condenser) shall be chloride free when tested with a small amount of deionized water.

5.2 Roll a rectangular (2" x 4") piece of the aluminum foil so that it will pass through the ground glass opening of the flask. Handle the foil with clean tweezers from now on.

5.3 Wash the aluminum foil scrupulously in acetone, deionized water, benzene and chloride free ether. Dry in an oven at 100°C for 1/2 hour.

5.4 Weigh the foil to the fourth decimal place, then place the foil in the flask. Make sure that none of the surfaces are touching.

5.5 Add 200 ml of the oil sample. Attach the condenser to the flask and shield all glassware from daylight.

5.6 Place the flask in the bath or on the heating mantle. The temperature of the oil under test shall be maintained at 210°C ± 5°C for 6 hrs. ± 1/4 hr.

5.7 At the end of the heating period, remove the foil and wash and weigh the foil as outlined in paras 5.3 and 5.4.

5.8 Record the initial and final color and acidity of the oil.

5.9 Check for chlorides. Final color, condition, and acidity should not exceed maximum values given on the individual part number prints.

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1.0 Scope

1.1 This section covers the method of testing for the resistivity of askarels and other related materials.

2.0 Definition.

2.1 Volume Resistivity

2.1.1 The volume resistivity of a material is the ratio of the potential gradient parallel to the current in the material to the current density.

2.2 Surface resistivity

2.2.1 The surface resistivity of a material is the ratio of the potential gradient parallel to the current along its surface to the current per unit width of the surface.

3.0 Significance

3.1 Volume and surface resistivity

3.1.1 When the volume and surface resistivities of an insulating material have been determined, it becomes possible to design an insulator for a specific application. The changes of both resistivities with temperature and humidity may be great, and must be known in order to meet operating conditions. Surface resistivity changes very rapidly with humidity, while volume resistivity changes only slowly, although the final change may be greater. Volume and surface resistivity measurements are often used in checking the uniformity of an insulating material, either to determine the uniformity of processing or to detect traces of impurities that affect the quality of the material and that may not be readily detectable by other methods.

4.0 Correlation

4.1 When adequate correlating data are available, resistivity may be used to indicate the suitability of a material in other respects, such as, dielectric breakdown, dissipation factor, moisture content, degree of cure, mechanical continuity, and deterioration from whatever cause. While the actual value of resistivity may be important initially for any material, it is the change in resistivity that is significant when there is a change in the material. The resistivity test is of greatest value when the test specimen has the same form as that required in actual use.

5.0 Method of test

5.1 Test for resistivity shall be performed in accordance with Aerovox TS-1, latest revision, pertinent paragraphs

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1.0 Scope

1.1 This section covers the method of testing for the Dielectric Constant of askarels and other related materials.

2.0 Definition

2.1 The dielectric constant, K, of an askarel is the ratio of the parallel capacitance (Cp) of a given configuration of electrodes with the askarel as the dielectric, to the capacitance (Ca) of the same electrode configuration with air as the dielectric:

$$K = \frac{C_p}{C_a}$$

3.0 Significance

3.1 The dielectric constant of an askarel is a measure of its relative ability to store electrostatic energy. It is of considerable importance in the design of capacitors and may also be useful as an indication of the composition and purity of the askarel.

4.0 Method of test

4.1 Test for dielectric constant shall be performed in accordance with Aerovox TS-1, latest revision, pertinent paragraphs.

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1.0 Scope

1.1 This section covers the method of testing for the dissipation Factor of Askarels and other related materials.

2.0 Definition

2.1 The dissipation factor of an askarel is the ratio of the conductance (G) of a capacitor in which the material is the dielectric to its susceptance (B); or the ratio of its parallel reactance (Xp) to its parallel resistance. It is also the tangent of the loss angle (tan 0 (also called loss tangent), and the cotangent of the phase angle (cot 0). It is represented by the symbol D:

$$D = \cot 0 = \tan 0 = \frac{G}{B} = \frac{Xp}{Rp}$$

$$= \frac{G}{WCp} = \frac{1}{RpWCp}$$

where: $W = 2\pi$ times the frequency in cycles per second

3.0 Significance

3.1 When adequate correlating data is available, dissipation factor may be used to indicate the suitability of a material in other respects, such as, dielectric breakdown, moisture content, degree of cure, and deterioration from whatever cause. While the actual value of dissipation factor is important initially for any material, it is the change in dissipation factor that is significant when there is a change in the material.

4.0 Method of test

4.1 Test for dissipation factor shall be performed in accordance with Aerovox TS-1, latest revision, pertinent paragraphs.

1.0 Scope

1.1 This method, under controlled conditions, indicates the relative thermal stability of chlorinated aromatic dielectric fluids, as well as the thermal instability resulting from possible contaminants. Two procedures are indicated as follows:

1.1.1 Procedure A, heating for 16 hours and

1.1.2 Procedure B, heating for 6 hours

1.2 The presence of compounds containing halogen other than chlorine, that is, bromine and iodine, capable of being liberated by exposure to the temperature employed in the test, will give positive results. Also, any other volatile foreign substance capable of reacting with silver nitrate will give positive results. Such materials would not normally be present in an askarel.

1.3 Askarels containing scavengers cannot be analyzed by this method because the scavenger would not allow the hydrogen chloride to be evolved.

2.0 Summary of method

2.1 The method employs potentiometric titration with silver nitrate in a limited aqueous medium to measure chloride ions obtained after heating unstable chlorine compounds in chlorinated aromatic hydro-carbons of the askarel dielectric class for 6 and 16 hours at 210°C in the presence of a regulated stream of pure air. While the purpose of the air stream is to sweep the volatile chlorides from the reaction flask into the absorber vessel where they are collected in a small amount of water, it is indicated that oxygen in the air stream induces the liberation of chlorides when askarels are heated at relatively high temperatures.

3.0 Apparatus

3.1 Heating bath (See Exhibit #2): A noncorrosive and insulated open bath fitted with adequate electric heating elements and controls capable of heating the bath fluid to 210°C and maintaining this temperature within ± 0.5°C throughout the entire bath, to avoid hot spots, is required. The bath should be placed in a hood. For most accurate results the test should be carried out in the absence of light, especially daylight and fluorescent light.

3.2 Bath fluid: Dow-Corning 550 Silicone has been found satisfactory for this purpose.

3.3 Air supply (See Exhibit #1): The air supply may be from a cylinder of "breathing air" such as used by skin divers or compressed air available from the plant or laboratory. The air shall be purified by passing successively through a scrubber bottle containing 40% sodium hydroxide (NaOH) to remove chlorides, an empty safety bottle, a bottle containing concentrated sulfuric acid (H_2SO_4) to remove ammonia, and finally a trap immersed in dry ice and acetone to remove possible organic impurities and moisture. This air-purifying train shall be cleaned periodically.

3.4 Air pressure regulators, for the required accurate regulation of air pressure. An additional regulator may be required if the pressure of the incoming air is above 5 psi (see Exhibit #1).

3.4.1 A model 40-2 regulator, 0 to 50 in. of water pressure, Moore Products Co., H and Lycoming Sts., Philadelphia, Pennsylvania, has been found satisfactory for this purpose.

3.5 Manifold and capillaries (See Exhibit #1): The purified air shall be connected to a glass manifold having one connection for each sample. Capillaries of the appropriate length shall be connected with gum rubber tubing between the manifold and the 8-mm glass tube inlet of the sample flask. The desired air flow through each sample flask is about 50 ml per min. This constant air flow is accomplished by providing a constant pressure on the manifold with suitable restriction in the form of the capillaries of suitable length between the manifold and the sample flasks.

3.6 Glass apparatus

3.6.1 Container for the sample placed into the heating bath shall be a 300-ml Erlenmeyer borosilicate glass flask with a standard-taper ground-glass joint. A 6 to 8-mm glass air inlet tube sealed into the standard-taper male ground-glass joint shall extend to near the bottom of the flask (see Exhibit #3).

3.6.2 Air outlet arm shall also be 6 to 8-mm glass tubing, sealed into the standard-taper male joint. The all-glass outlet tubing is designed to allow the absorber flask to be positioned so that it will be at about room temperature.

3.6.3 Absorber flask shall be a 125-ml Volhard nitrogen bulb. A rubber stopper mounted on the end of the air outlet tube shall serve to connect this tube with the absorber flask. The conduit leading from the main part of this absorber flask into its attached bulb should be constructed so that the air will flow through and not merely over the water contained in the bulb (see Exhibit #4).

3.7 Magnetic stirrer and stirring bar: The stirrer shall be equipped with speed control. The bar shall be of one piece covered with tetrafluoroethylene (Teflon), (see Exhibit #2).

3.8 Microburet: 1.0-ml capacity and graduated in 0.01-ml divisions.

3.9 Electrodes: silver and standard glass.

3.9.1 Although the silver billet-glass electrode system is preferred, any other standard reference electrode may be used provided that there is no chloride contamination of the sample. However, a silver-silver chloride electrode system should not be used.

3.9.2 Beckman Catalog No. 39261 billet electrode and Catalog No. 40498 glass electrode have been found satisfactory for this purpose.

3.10 Potentiometer: Suitable for use with a glass electrode.

3.10.1 The Beckman G.S. or Zeromatic model and the Leeds and Northrup line pH meter have been found satisfactory for this purpose.

4.0 Reagents

4.1 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. 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.

4.2 Purity of water: Unless otherwise indicated, reference to water shall be understood to mean chloride-free reagent water conforming to the Specifications for Reagent Water (ASTM Designation: D-1193).

4.3 Acetone, chloride-free: If the acetone is contaminated with chlorides, it shall be distilled over silver nitrate.

4.4 Nitric acid (1:100): Dilute 1 part of concentrated nitric acid (HNO_3) in 100 parts of water.

4.5 Silver nitrate, standard solution (0.0025 N): Dissolve 0.4247 g of silver nitrate ($AgNO_3$) in water, transfer it into a 1-liter volumetric flask, add 3.0 ml of concentrated nitric acid (HNO_3), and dilute to volume. Standardize this solution against a pure chloride standard. Check the solution at least monthly to assure a consistent reagent.

4.5.1 If a standard 0.1 N $AgNO_3$ solution is available, suitable dilution may be made.

5.0 Procedure A, Heating for 16 hours

5.1 Because of the very small amount of chloride in the normal askarels extreme care must be taken to minimize contamination.

5.2 Weigh 290 $\pm$ 1 g of the sample into the 300-ml Erlenmeyer flask. Connect the ground-glass joint containing the air inlet and the air outlet tube, and place the flask into the heating bath at 210° $\pm$ 0.5°C. The bath fluid level should be about 1 inch below the bottom of the ground-glass joint of the flask.

5.3 Place 20 ml of water into the Volhard flask absorber and attach it to the rubber stopper on the air outlet tube from the Erlenmeyer flask.

5.4 Connect the purified air supply from the capillary to the inlet of the Erlenmeyer flask. Adjust the air pressure regulator to give the pressure needed to give the desired air flow, about 40 to 50 ml per min. Keep the sample in the dark during the 16 hour heating period.

5.5 When the heating has been completed, transfer the water from the absorber into a 200-ml beaker, using 160 ml of chloride-free acetone to effect quantitative transfer.

5.6 Add 4 drops of HNO_3 (1:100).

NOTICE

5.7 Place the beaker on the magnetic stirrer and stir the sample. Insert the silver billet glass electrode system and titrate with 0.0025 N A_gNO_3 solution, using the microburet. Normal samples of askarel require extremely small amounts of 0.0025 N A_gNO_3 solution. Therefore, when titrating use 0.01-ml additions of the solution and allow sufficient time for equilibrium to be established before recording the emf change. If three or four additions of 0.01-ml increments of 0.0025 N A_gNO_3 solution give a potential change of less than 1 mv per 0.01 ml of solution, add larger amounts such as 0.05 ml until such a change is observed. Then reduce the additions again to 0.01 ml to complete the titration. The end point is the point where the greatest potential change per 0.01-ml increment of 0.0025 N A_gNO_3 solution is obtained.

5.8 Blank: Carry out the entire procedure again except for omission of the sample in order to obtain an over-all blank.

5.8.1 The amount of chloride ions measured when the test is applied to normally stable askarel is very small, less than 1 ppm. Therefore, the test must be made in an area where the atmosphere is free from contaminating influences, and it is essential to make a simultaneous blank determination. A normal blank is about 0.03 ppm of chlorides. The value obtained on the blank will indicate the degree to which the test environment, reagents, and glassware are satisfactory for this type of testing.

6.0 Procedure B, Heating for 6 hours

6.1 Run the 6 hour thermal test exactly as the 16 hour test except for the difference in the time. Results are roughly one half of the total obtained from the 16 hour test.

7.0 Calculation

7.1 Calculate the reactive chlorine as follows:

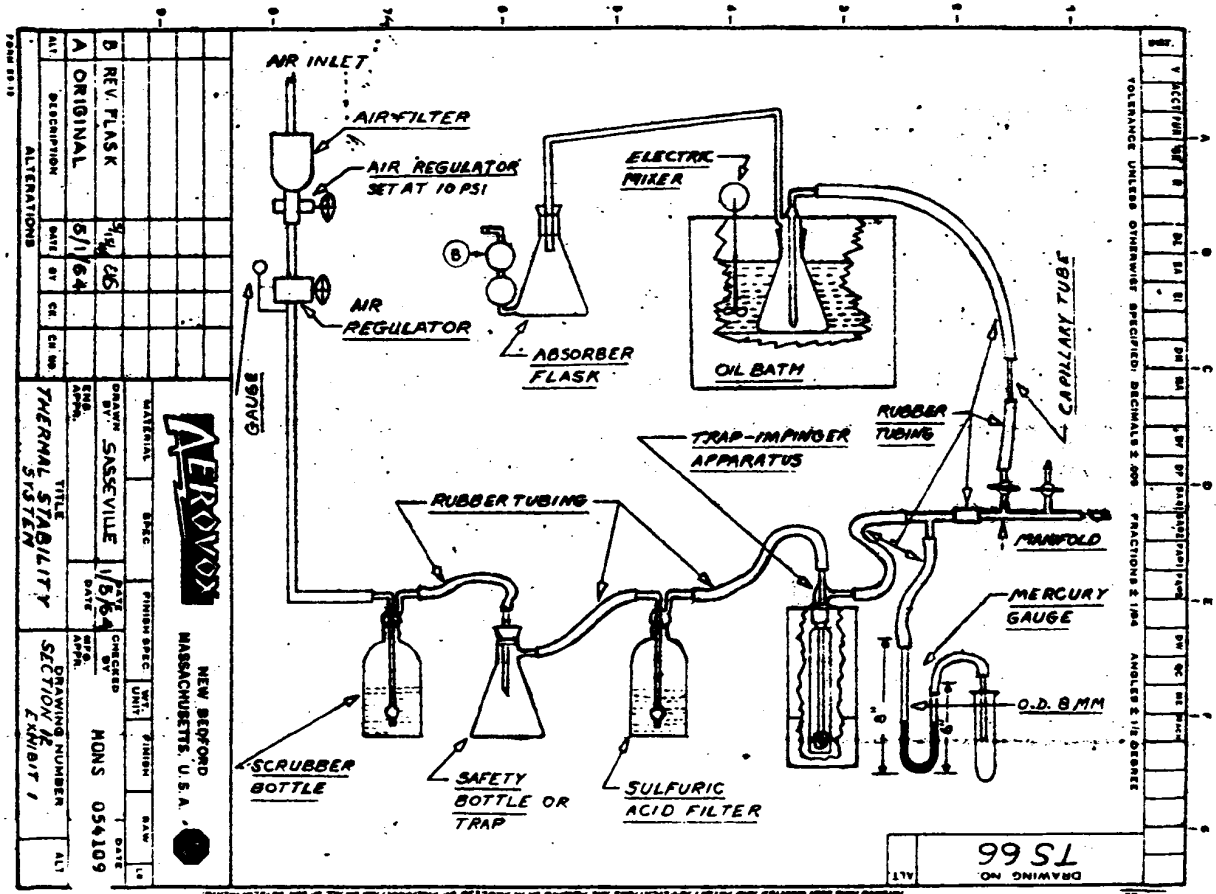
$$\text{Reactive chlorine, ppm} = \frac{(A - B)\ N \times 35.46}{W} \times 1000$$

where:

A = milliliters of A_gNO_3 solution required for titration of the sample,
B = milliliters of A_gNO_3 solution required for titration of the blank,
N = normality of the A_gNO_3 solution, and
W = grams of sample used.

8.0 Reproducibility

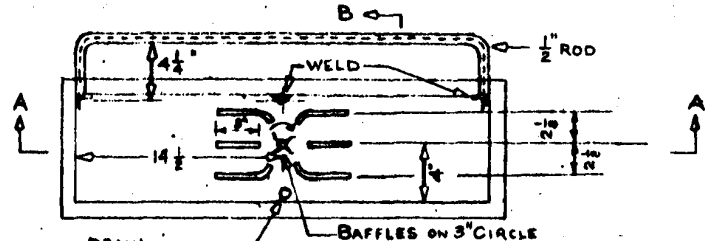
8.1 This is not a precise analytical procedure. If the required precautions and care are taken, it is possible to obtain reasonable reproducibility, provided that the chloride level does not exceed about 1 ppm. At higher chloride levels good reproducibility cannot be expected.


AIR INLET
AIR FILTER
AIR REGULATOR SET AT 10 PSI
AIR REGULATOR
GAUGE
ABSORBER FLASK
ELECTRIC MIXER
OIL BATH
CAPILLARY TUBE
RUBBER TUBING
TRAP-IMPINGER APPARATUS
RUBBER TUBING
MANIFOLD
MERCURY GAUGE
O.D. 8 MM
SCRUBBER BOTTLE
SAFETY BOTTLE OR TRAP
SULFURIC ACID FILTER
TOLERANCE UNLESS OTHERWISE SPECIFIED: DECIMALS ± .005 FRACTIONS ± 1/64 ANGLES ± 1/2 DEGREE
DRAWING NO. TS 66
ALT
B REV. FLASK
A ORIGINAL
ALT. DESCRIPTION DATE BY CK. CH. NO.
ALTERATIONS
AEROVOX
NEW BEDFORD MASSACHUSETTS, U.S.A.
MATERIAL SPEC. FINISH SPEC. WT. UNIT FINISH RAW LB
DRAWN BY SASSEVILLE DATE 1/5/64
ENG. APPR. DATE
CHECKED BY MFG. APPR.
TITLE THERMAL STABILITY SYSTEM
DRAWING NUMBER SECTION 12 EXHIBIT 1
MONS 054109
ALT
FORM 59-12

TOLERANCE UNLESS OTHERWISE SPECIFIED: DECIMALS ± .005 FRACTIONS ± 1/64 ANGLES ± 1/2 DEGREE

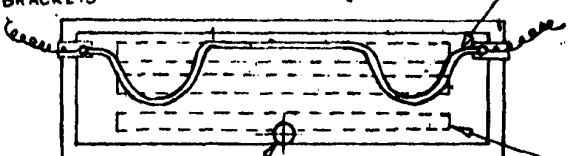
DRAWING NO. TS66 ALT

TOP VIEW



HEATER BRACKETS

500 WATT LO-LAG HEATER. AMIN CO. 110 V.



TOP VIEW SHOWING HEATERS

3 500 WATT HOV GE STRIP HEATERS, BOLT TO BOTTOM OF BATH & WELD AROUND HEAD OF BOLT TO MAKE LEAKPROOF.

THERMO-REGULATOR CENCO INCENTER

ALT.	DESCRIPTION	DATE	BY	CK.	CR. NO.
B	REDRAWN NO CHANGE	5/1/64			
A	ORIGINAL	9/31/63			

ALTERATIONS

AEROVOX NEW BEDFORD MASSACHUSETTS, U.S.A.

MATERIAL	SPEC.	FINISH SPEC.	WT. UNIT	FINISH	RAW	LB

DRAWN BY SASSEVILLE DATE 5/1/64 CHECKED BY

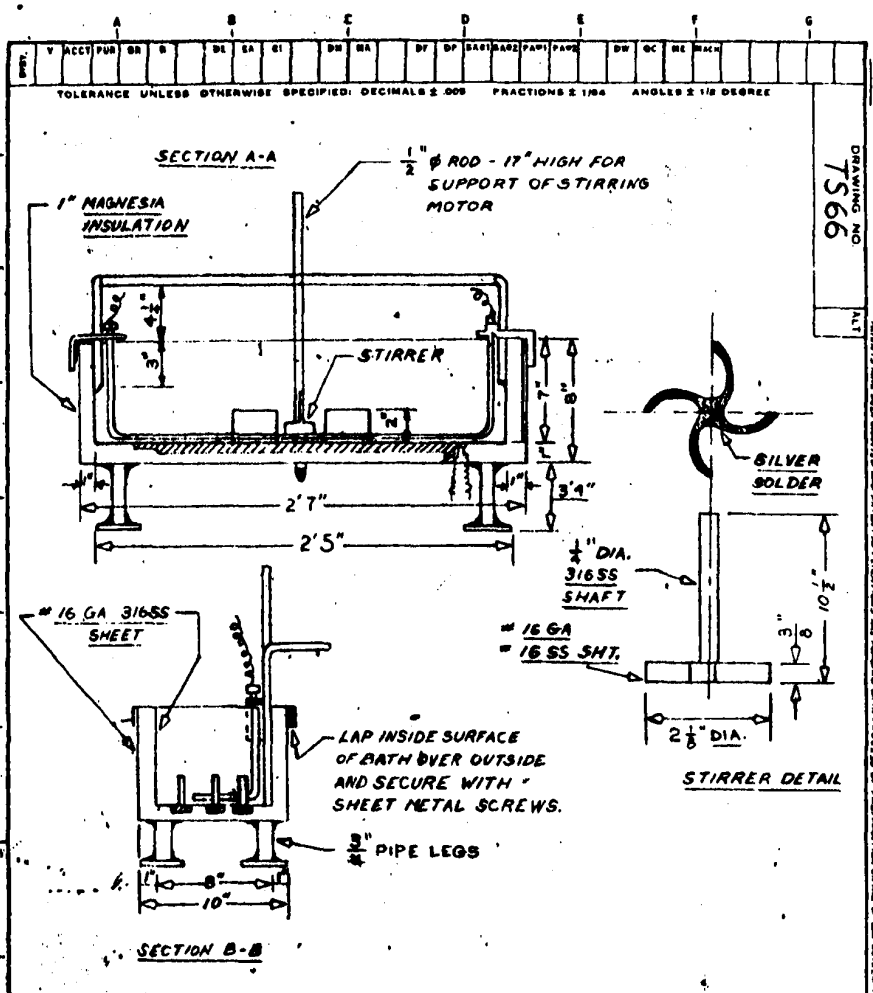
ENG. APPR. DATE MFG. APPR.

MONS 054110

TITLE OIL BATH SHEET 2

DRAWING NUMBER SECTION 12 EXHIBIT 2 ALT

FORM ED-10



DRAWING NO. TS66

ALT

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AEROVOX NEW BEDFORD MASSACHUSETTS, U.S.A.

MATERIAL	SPEC.	FINISH SPEC.	WT. UNIT	FINISH	RAW	LB

ALT.	DESCRIPTION	DATE	BY	CK.	CH NO.
B	REDRAWN NO CHANGE	5/1/64	CHS		
A	ORIGINAL	11/10/63			

ALTERATIONS

DRAWN BY SASSEVILLE DATE 5/1/64 CHECKED BY

ENG. APPR. DATE MFG. APPR.

TITLE OIL BATH SHEET 2

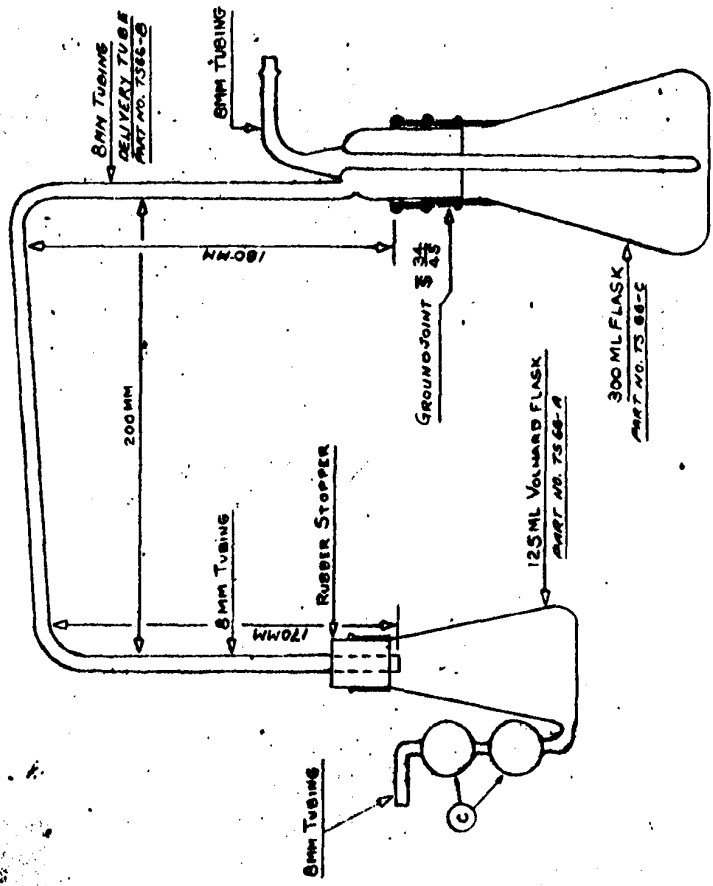
DRAWING NUMBER SECTION 12 EXHIBIT 2 ALT

MONS 054111

FORM 20 12

TOLERANCE UNLESS OTHERWISE SPECIFIED: DECIMALS ± .005 FRACTIONS ± 1/64 ANGLES ± 1/2 DEGREE

DRAWING NO. TS66



ALT.	DESCRIPTION	DATE	BY	CK.	CH NO.
C	REV FLASK	4/14/64	EWS		
B	REDRAWN NO CHANGE	5/1/64	EWS		
A	ORIGINAL	3/10/57			

ALTERATIONS

AEROVOX

NEW BEDFORD
MASSACHUSETTS. U.S.A.

MATERIAL	SPEC	FINISH SPEC	WT. UNIT	FINISH	RAW	LB

DRAWN BY SASSEVILLE. DATE 5/1/64 CHECKED BY MONS 054112

ENG. APPR. DATE MFG. APPR. DATE

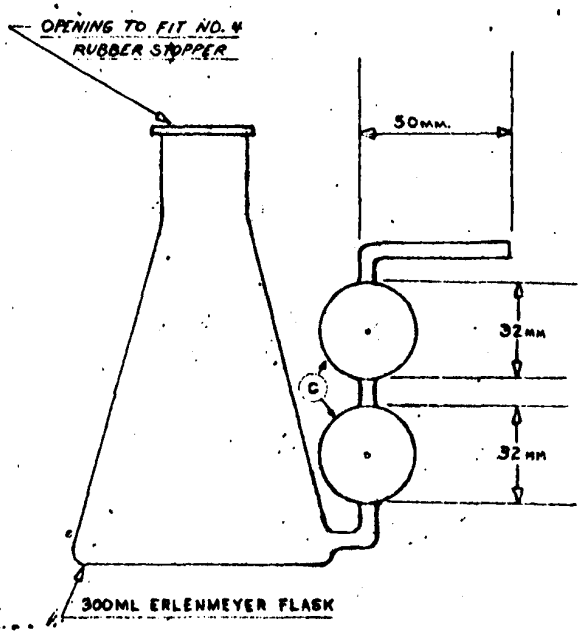
TITLE

DRAWING NUMBER SECTION 12 EXHIBIT B ALT

FORM 20-12

TOLERANCE UNLESS OTHERWISE SPECIFIED: DECIMALS ± .005 FRACTIONS ± 1/64 ANGLES ± 1/2 DEGREE

DRAWING NO. TS 66 ALT



AEROVOX NEW BEDFORD MASSACHUSETTS, U.S.A.

MATERIAL	SPEC.	FINISH SPEC	WT. UNIT	FINISH	RAW	LS

DRAWN BY SASSEVILLE EM 3/30/64 CHECKED BY MONS 054113

END. APPR. DATE MFG. APPR.

TITLE FLASK

DRAWING NUMBER SECTION 12 EXHIBIT 4 ALT

ALT.	DESCRIPTION	DATE	BY	CK.	CH. NO.
D	REV. BULB				

* ALTERATIONS

FORM 20-12

1.0 Scope

1.1 This method covers the determination of Hydrolyzable chlorine compounds in askarels and other related products.

2.0 Summary of method

2.1 This method is a quantitative potentiometric titration using silver nitrate in an essentially non-aqueous medium to determine chloride ions obtained after methanolic sodium hydroxide hydrolysis of unstable chlorine compounds in chlorinated aromatic hydrocarbons of the askarel dielectric class.

3.0 Significance

3.1 The measured chloride ion, reported as parts per million in the askarel sample, is indicative of the relative stability of the askarel under prescribed conditions of test.

4.0 Interferences

4.1 The presence of compounds containing hydrolyzable halogen other than chlorine, that is, bromine and iodine, will give positive results, as will also any other foreign substance capable of forming an insoluble compound with silver nitrate. Such materials would not normally be present in an askarel.

4.2 The amount of chloride ions measured when the test is applied to normally stable askarels is very small, ranging from 0.1 to a few parts per million. Therefore, the test must be made in an area where the atmosphere is free from contaminating influences, and it is essential to make a simultaneous blank determination. The value obtained on the blank will indicate the degree to which the test environment, reagents, and glassware are satisfactory for this type of testing.

5.0 Apparatus

5.1 pH assembly — Leeds and Northrup Catalog No. 7664-A-1

5.2 Glass electrode — Beckman Catalog No. 40498

5.3 Silver Electrode — Beckman Catalog No. 39261

5.4 Flask, Erlenmeyer, T 24/40, 500 ml capacity

5.5 Glass Condenser, T 24/40 Tip, 400 mm Jacket

5.6 Hot Plate Magnetic Stirrer. Sargent Catalog No. S-40990

5.7 Teflon covered magnetic stirring bar

5.8 Micro Buret graduated in 0.01 ml divisions
Scientific Glass Apparatus Co., Catalog No. JM-570

5.9 Usual laboratory glassware.

6.0 Reagents

6.1 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. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

6.2 Purity of water: unless otherwise indicated, references to water shall be understood to mean reagent water conforming to the specifications for reagent water (ASTM D-1193). In addition, the water shall be chloride-free, as detected by this test.

6.3 Methanol (chloride free): five liters of methanol are refluxed with 0.5 grams of analytical reagent grade silver nitrate ($AgNO_3$) for 1/2 hour. The methanol is then distilled from the $AgNO_3$, discarding the first 100 ml to flush the apparatus. 90% of the charge is distilled from the flask and the contents of the flask are discarded. The methanol should be checked to assure purity by titration. The chloride ion concentration should be less than 0.02 ml of 0.0025 N $AgNO_3$ per 100 ml of methanol.

6.4 Sodium hydroxide reagent: analytical reagent grade sodium hydroxide (NaOH) is used to prepare this reagent. A 0.1N solution is prepared by dissolving 4.0 grams of analytical reagent grade NaOH in 1 liter of chloride free methanol.

6.5 Sulfuric Acid: prepared by a 50:50 volumetric dilution of analytical reagent grade concentrated sulfuric acid with chloride free distilled water. The acid is always poured into the water with constant stirring to prevent any dangerous build-up of heat.

6.6 0.0025 N $AgNO_3$: dissolve 0.4247 grams of analytical reagent grade $AgNO_3$ crystals in one liter of chloride free water containing 3.0 ml of concentrated nitric acid. This solution should be standardized against a pure chloride standard. A sodium chloride crystal such as used in infra-red spectrometer cells is a good source of pure NaCL. The $AgNO_3$ solutions should be checked (at least monthly) to assure a consistent reagent.

6.7 Acetone (chloride free): prepared by distillation from $AgNO_3$ as described above for methanol and should also be checked by potentiometric titration to assure optimum purity. Normally a chloride content of less than 0.02 ml of 0.0025 N $AgNO_3$ per 100 ml is derived by this method.

7.0 Procedure

7.1 Tare a 500 ml flask to the nearest 0.1 gram, using a trip balance.

7.2 Mix the sample and pour a small amount over the lip of the sample bottle to clean it.

7.3 Add 100 ± 1 gram of sample to the flask. Reweigh to obtain weight of sample.

7.4 Using clean dry tongs, place the magnetic stirring bar in the flask.

7.5 Add, by graduate, 100 ml of 0.1 N methanolic NaOH.

7.6 Place the flask on a hot plate-stirrer and attach condenser. Stir gently and allow to reflux for one hour from start of reflux.

7.7 Turn off heat and place container of cold water under the flask to cool sample. Rinse down the condenser with chloride free acetone and quantitatively transfer the solution to a 400 ml beaker using a total of 100 ml of acetone.

7.8 Acidify by adding 3 ml of 1:1 sulfuric acid, place on a magnetic stirrer under the silver-glass electrode system.

7.9 Titrate the sample to largest change in emf using 0.0025 N $AgNO_3$. The titrant is added in 0.10 ml increments until a change in emf of 1 mv or greater is noted, then proceed in 0.05 ml increments.

7.10 Run a reagent blank exactly as above omitting the sample.

7.11 Calculation

$$\text{Reactive Chlorine (ppm)} = \frac{(\text{Sample Titer-Blank Titer})\ (\text{N } AgNO_3)\ (35.46)\ (10^3)}{\text{Sample Weight}}$$

or for 100 gram sample and 0.0025 N $AgNO_3$

Reactive Chlorine (ppm) = (Sample Titer-Blank Titer) (0.886)

NOTE: All glassware and equipment must be scrupulously clean and free of chlorides. Glassware may be cleaned with hot dilute nitric acid; rinse with distilled water and then chloride free acetone, to speed drying. If the titer for the reagent blank is above 0.6 ml of 0.0025 N $AgNO_3$, probably the reagents are contaminated or glassware and electrodes are not properly cleaned.

References: 1. Anniston Method 14-62-57 and 092
2. Westinghouse Specification 80138, dated 11-4-60.

Issue Record

Issue	Revisions	Chief Engr	Tech Writer	Stds Mgr	Date
A	Original				3 Oct 50
B	Rev & Rewr				31 May 57
C	Added #11-Thermal Stability				27 Aug 57
D	Rev pges 1 & 12				16 June 58
E	Rev 3(1) & 8(B)				8 May 59
F	Rewritten	A.Kalstein	R.Bodner	T.P.Richards	26 Apr 63
G	Completely sectionalized spec. Rev. Sections 5,7 & 12	A Kalstein	E Horvath	T P Richards	17 Nov 64

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