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IPC-TM-650
TEST METHODS MANUAL

Number **2.3.25**	
Subject **Detection and Measurement of Ionizable Surface Contaminants**	
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Originating Task Group **Ionic Conductivity TG (5-32a)**	

1.0 Scope

1.1 Purpose These tests are designed to indicate the presence and amounts of ionic surface soils on a printed board and printed board assemblies, or components and that are soluble in the test solution. Bulk ionic cleanliness testing may be accomplished by three methods: 1) Dynamic extraction method; 2) Static extraction method; 3) Resistivity of Solvent Extract (ROSE) method.

1.2 Restrictions Measurements of ionic conductivities do not differentiate between different ionic species. They simply measure conductivities (or resistivities) which can be related to amounts of ionic materials present in solution. There is no identification of the contribution to the total conductivity readings of any individual ionic species which may be present in the solution. For simplicity, amounts of ionic materials in solution can be expressed by a conductivity factor which is equivalent to the measurement conductivity contributed by a known amount of a standard strongly ionized salt such as sodium chloride (NaCl). Ionic residues are therefore usually expressed as equivalents of sodium chloride in micrograms per unit surface area of the sample. This does not imply that the contamination is NaCl but, rather, it exhibits a conductivity function which is equivalent to that of the expressed amount of sodium chloride if it were in solution instead of the ionic soil.

These tests will not measure any surface ionic materials which are not brought into solution because of insolubility, physical entrapment or inadequate exposure to the extracting solvent. Additionally, non-ionic components of the soil will not be measured.

1.3 Application Correlation between test equipment must be established or required for comparison purposes. These methods are applicable as quality control tools in evaluating the effective parameters of the materials used and the cleaning process in terms of how they affect the final cleanliness of the board or assembly. As process control tools, they can be used to inspect printed wiring boards or printed board assemblies and determine if they conform to the cleanliness level requirements of the user's performance specification. In process development, these procedures can be used to evaluate flux cleanability, solvent efficiency and optimization of process parameters.

2.0 Applicable Documents

2.1 See 7.1 References

3.0 Test Specimen Any printed wiring board or assembly of sufficient area to provide enough solvent sample to determine its resistivity.

4.0 Resistance of Solvent Extract Method

4.1 Description The original Resistance of Solvent Extract (ROSE) method utilized the manual extraction of ionic material from a sample surface. This consisted of flushing the surface with a stream of 2-propanol/water mixture and carefully catching all of the drippings before measuring the resistivity of the composite sample.

4.2 Test Equipment and Chemicals

4.2.1 Miscellaneous laboratory ware (e.g., beakers, funnels, storage bottles and graduated cylinders). This plastic ware can be high density polyethylene, polmethylpentene (polypentene) or equivalent. Glassware cannot be used because it has been shown to contribute ionic contamination in a short time with this solvent/water solution.

4.2.2 Conductivity Bridge and liquid conductivity cell apparatus capable of measuring specific resistances within a range covering at least 100Kohm-cm to 20 Mohm-cm.

4.2.3 Deionizing Column Barnstead HN Ultrapure Mixed Bed or equivalent. *NOTE:* Some of these columns are color dyed. This dye will interfere with test results. Make certain that the column used has no dye.

4.2.4 Wash solution composed of 75% v/v 2-propanol/water or 50% v/v 2-propanol/water. This wash solution must be deionized to a resistance equal to or greater than 6 X 10^6 ohms-cm (conductivity less than 0.166 microsiemens/cm) If stored, this wash solution must be freshly deionized prior to use. Typical resistivity of 25 X 10^6 ohm-cm (conductivity of 0.04 microsiemens/cm) can be achieved and is preferred to reduce the deadband zone.

4.3 Calibration of Bridge This is essential in the ROSE method because there can be no correlation between resistivity/conductivity readings and NaCl equivalents without calibration. All future specification requirements are to be stated in maximum micrograms/cm^2 NaCl equivalents.

4.3.1 Prepare a standard NaCl solution from a weight of reagent grade NaCl salt dissolved in deionized water to produce a final diluted concentration of 0.06 grams/liter NaCl (5 ml equals 300 micrograms NaCl).

4.3.2 Place one liter of the 2-propanol water solution (25±2°C or the reference temperature of the bridge in use) in a plastic beaker.

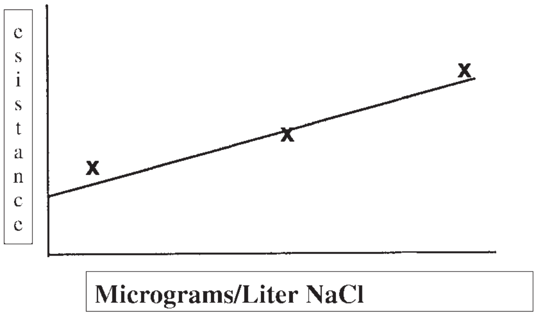
NOTE: The 75% v/v or 50% v/v 2-propanol solution must be used in this calibration. Water cannot be used since it is not the test solution used in the procedure. The test solution used in this calibration can be recleaned by passing through the DI column until the required resistivity/conductivity is obtained.

4.3.3 From a 50 ml burette, add to the liter of test solution, 5 ml of the standard 0.06 grams/liter NaCl solution. Stir and measure resistance/conductivity.

4.3.4 From a 50 ml burette, add to the liter of test solution, 20 additional ml of the standard 0.06 grams/liter NaCl solution, for a total of 25 ml. Stir and measure resistance/conductivity.

4.3.5 From a 50 ml burette, add to the liter of test solution, 25 additional ml of the standard 0.06 grams/liter NaCl solution, for a total of 50 ml. Stir and measure resistance/conductivity.

4.3.6 Plot a three point nomogram Resistance/Conductivity vs Micrograms NaCl/liter.



If a multi-range meter is used the curve should not be extended beyond the maximum reading of the meter for that range, unless linearity is proven by additional points obtained by adding more standard salt solution. The nomogram will never cross the zero point of resistance/conductivity.

4.4 Test Procedure

4.4.1 Carefully preclean all plastic ware with deionized water followed by a final rinse with the 2-propanol test solution.

4.4.2 Determine the surface area of the printed wiring board or assembly, including both sides. Subtract an estimated value for any cut-out section of the board.

4.4.3 Suspend the test specimen within a convenient sized funnel positioned over a graduated cylinder. Care must be taken not to handle the sample or any of the appliances used to hold the sample with bare hands.

4.4.4 Direct a fine stream of freshly-deionized test solution on both sides of the specimen, covering all board and component surfaces. Continue this process, slowly collecting all the runoff in the graduated cylinder until a volume of the 2-propanol/water mixture has been collected. Use 10ml/650 cm^2 (10ml/in^2) of board area. The volume collected is not critical, but the total volume must be exactly recorded. A volume correction is made in the calculation.

4.4.5 Pour the final measured volume into a polypentene beaker, stir and measure the resistivity/conductivity with the bridge probe.

4.4.6 The resistivity/conductivity readings can be used to convert the micrograms of NaCl equivalents as follows:

1. Locate where the resistivity or conductivity, intersects the calibration curve on the X-axis
2. Extend a vertical line from the point of intersection to the x-axis. Read and record micrograms/liter NaCl (M).
3. Multiply the concentration in micrograms/liter NaCl by the total liters of test solution used (V). This result indicates the total micrograms of NaCl equivalents removed from the printed wiring board (T).
4. Divide the micrograms of NaCl equivalents by the area of the printed wiring board or assembly (A). This yields the micrograms of NaCl equivalents per square centimeters or square inch.

SAMPLE CALCULATION:

M X V = T; T/A = micrograms NaCl/sq cm

5.0 Dynamic Extraction Method

5.1 Description In the dynamic method, a purified alcohol/water mixture is circulated into and out of a test tank chamber containing the sample being tested. The mixture exiting the test tank is passed through a conductivity cell which measures the conductivity continuously. These conductivity values are integrated over the time of the extraction. The mixture is then pumped through a resin deionization column before it is recirculated back into the test tank. As ionic materials are extracted from the samples and then pumped out of the cell, the conductivity of the solution will change dynamically until all of the extractable ionic material has been removed.

5.2 Test Equipment A dynamic conductivity measurement system including a test tank, a temperature compensated conductivity cell, ion exchange columns and a metering pump connected together in a recirculating loop as described in 5.1. The conductivity readings are integrated over the time of the measurement by electronic integration. The equipment may have the capability of heating the alcohol/water mixture to accelerate extraction of ionic soils from poorly accessible places such as under surface mounted components.

5.3 Procedure

5.3.1 Solvent Systems Industry has established two different standard test solutions that are used worldwide:

75% v/v 2-propanol/water
50% v/v 2-propanol/water

Select the solution required by your specification (e.g., J-STD, engineering drawing specifications, contract documentation, etc.).

5.3.2 Calibration Once the fluid in the system has established a stable level of conductivity, a precise quantity of a sodium chloride calibration solution is injected into the test solution in the test tank. This is done according to the verification of calibration instructions provided by the manufacturer of the equipment manufacturer of the equipment being used.

5.3.3 Testing Once the system has been calibrated or verified in accordance with 5.3.2 and a stable baseline has been achieved, the test specimen is immersed into the sample tank. Care must be taken not to handle the sample or any of the appliances used to insert it into the tank. Finger dirt contains ionic soils which may contribute to spurious readings.

During the course of the measurement, the conductivity will rise from the initial baseline level and then gradually return. When it has returned to the baseline level, no additional ionic material can be removed and the measurement is complete.

5.4 Interpretation of Test Data The number obtained from this type of measurement indicates the total amount of ionic material extracted from the entire sample in terms of equivalent amounts of sodium chloride (assuming the calibration was done with sodium chloride). This should be divided by the total surface area of the sample from which the ions were extracted to determine the surface ionic density of the original sample. For circuit board assemblies, it is common practice to use the total area of both sided of the printed board plus the total area of the components on the board.

The actual surface ionic density is most commonly calculated by programming this area into the instrument's microprocessor system. The total ionic amount will then be automatically divided by the area to indicate surface ionic density in terms of micrograms of sodium chloride per unit of surface are:

µg./cm^{2}eq.NaCl or µg./in^{2}eq.NaCl

6.0 Static Extraction Method

6.1 Description In the static extraction method, a measured volume of freshly deionized alcohol/water mixture is introduced into the test tank and its resistivity (or conductivity) measured continuously while the alcohol/water mixture is agitated. The board or assembly is then introduced into the solvent mixture to extract any soluble ionic surface contamination into solution. The resistivity (or conductivity) is monitored continuously until no further change with time is observed. This indicates complete removal of all of the available ionic soil. Since all of the ionic material is accumulated in a fixed (or static) amount of the extracting solvent mixture, the final reading is indicative of the total soluble ionic material extracted from the sample.

After the test is completed the solvent mixture is passed through ion exchange columns to remove ionic materials and regenerate the alcohol/water solvent mixture to its original high resistivity level for further tests.

6.2 Test Equipment A static conductivity measurement systems including a test tank, a temperature-compensated conductivity cell and monitor, means for solution agitation and a means for removing, deionizing and re-introducing the solvent mixture into the test tank before a new test is started. The equipment may also have the capability of heating the alcohol/water mixture to accelerate and improve the efficiency of extraction of ionic solid from poorly accessible regions, such as under surface-mounted components.

6.3 Solvent Systems See 5.3.1.

6.3.1 Calibration A precise quantity of sodium chloride calibration solution is injected into a designated volume of the test solvent mixture in the sample measurement cell. This is done according to the calibration or verification instructions provided by the manufacturer of the equipment being used.

6.3.2 Testing Once the system has been calibrated or verified in accordance with 6.3.1, the sample tank is filled as directed by the procedures of the equipment manufacturer and the test specimen is immersed in the tank. The minimum starting resistivity for this type of equipment is machine dependent. Ensure that the starting resistivity is below the ‘‘dead band zone’’ for equipment as defined in EMPF report RR0013. Care must be taken not to handle the sample or any of the appliances used to insert it into the tank. Finger dirt contains ionic solid which may contribute to spurious reading.

During the course of the measurement, the resistivity will fall continually as ionic material is extracted into solution. If conductivity is being monitored, it will be initially be very low, rising continually as ionic material is dissolved from the sample. The test can be terminated when there is no further change, in time, of the resistivity or conductivity functions. This can be established electronically in most commercially available equipment. The initial and final values together with the volume of the solvent mixture in the test tank, and sample surface area are used by the system to calculate the ionic levels which were present on the sample surface prior to the test.

6.3.3 Refer to the manufacturer’s equipment manual for optimal operation.

6.4 Treatment of Test Data See 5.4.1.

7.0 Notes

7.1 References Contact EMPF for copies of a report detailing comparative studies of cleanliness testing equipment.

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Methods of Measurement of Ionic
Surface Contamination
by Jack Brous
Available from Alpha Metals

Evaluation of Post-Solder Flux Removal
by Jack Brous
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