Association Connecting Electronics Industries

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IPC-TM-650
TEST METHODS MANUAL

Number	
2.3.28.2	
Subject	
Bare Printed Board Cleanliness by Ion Chromatography	
Date	Revision
12/2009	
Originating Task Group	
Ion Chromatography/Ionic Conductivity Task Group (5-32a)	

1 Scope This test procedure is designed to measure the level of anionic and cationic residues on the surface of unpopulated (bare) printed boards by ion chromatography.

2 Applicable Documents

IPC-WP-008 Setting Up Ion Chromatography Capability

3 Test Specimen Any printed board

4 Apparatus and Materials

4.1 Ion Chromatograph capable of accurately measuring ion concentration down to 0.5 parts per million (ppm). The equipment and chemistry should be set up and standardized per the manufacturer's instructions. The separation column and eluent composition should be chosen to provide good separation of the ions of interest.

4.2 Hot Water Bath capable of maintaining 80 ± 2 °C [176 ± 3.6 °F].

4.3 Clean extraction vessels.

4.4 Clean labware.

4.5 Clean gloves, e.g., cleanroom vinyl gloves. (<3 ppm of Chloride).

4.6 Deionized water with a starting resistivity of at least 16.0 megohm-centimeter.

4.7 HPLC or ACS grade chemicals for eluent and regenerant preparation.

4.8 National standard - traceable calibration standards (e.g., NIST traceable).

Note: At the time of publication of this test method, a traceable industry adipic acid standard does not exist. Future revisions to this test method will indicate such traceability when available.

4.9 2-Propanol (IPA), 99.5% ACS grade or better.

4.10 Volumetric Flasks (Typically 25, 50, 100, and 1000 ml)

4.11 Precision Pipetting Equipment (such as Eppendorf)

4.12 Ionics-free extract solution sample containers or auto-sampler vials if an auto-sampler is used with the instrument. Clean syringes should be used for manual injections.

5 Test Procedures

5.1 Extraction Procedure Select a low-ion extraction bag sized to fit the board with approx. 2.5 cm [1.0 in] excess on each side to minimize required extract solution, with several inches at the top to allow for air expansion when the bag is heated.

5.1.2 Use clean gloves when handling the samples to be tested. Place each sample in an extraction bag.

5.1.3 Prepare a 10%/90% volume/volume 2-propanol / deionized water solution for the extraction.

5.1.4 Add a known volume of the extraction solution to the extraction bag covering the printed board (approx. 0.5 mL/cm^2 of surface area).

5.1.5 Add the same volume of extraction solution to an empty bag of the same lot for use as a blank.

5.1.6 Suspend the bags into the 80 ± 2 °C [176 ± 3.6 °F] water bath allowing the water to force most of the air from the bags. Do not allow any of the water from the water bath into the extract solution in the bags. Fold the top of the bags over the suspending bar and clip in place with binder clips. This will minimize solvent loss during the extraction, yet not create a sealed bag. Alternatively, the bags may be heat sealed after forcing most of the air from the bag.

5.1.7 Allow the sample to soak for one hour (-0 min., +5 min.).

5.1.8 Remove the bags from the water bath and allow the bags/solution to cool to room temperature.

5.1.9 Shake the bag to mix the contents. Transfer solution to virgin sample vials for analysis or pull the sample solution directly from the bag using a clean syringe for manual injections.

5.1.10 Remove the test board from the bag, using gloved hands.

5.2 Calibration Procedure

5.2.1 The ions in the calibration procedure are a minimum. Other ions may be added to the determination if desired.

5.2.2 A part per million (ppm) is 1 milligram of the ionic species (solute, e.g., chloride ion) per 1000 grams of solution.

5.2.3 Prepare or purchase a combination stock anion standard solution: 5 ppm fluoride, 25 ppm acetate, 50 ppm formate, 20 ppm methanesulfonic acid (MSA), 50 ppm chloride, 50 ppm bromide, 20 ppm nitrate, 200 ppm adipate, 100 ppm succinate, 20 ppm sulfate, and 20 ppm phosphate.

5.2.3.1 This standard stock can be prepared from 1000 ppm stock solutions using the following formula:

Combine 0.125 ml fluoride; 0.5 ml MSA, nitrate, phosphate, and sulfate; 0.625 ml acetate; 1.25 ml formate, chloride, and bromide; 2.5 ml succinate; 5 ml adipate; in a 25 ml volumetric flask. Dilute to volume with 10% 2-propanol / 90% deionized water. The stock solution is stable for several weeks if kept refrigerated. Warm to room temperature before pipetting.

5.2.4 Prepare or purchase a combination stock cation standard solution: 5 ppm lithium, 50 ppm sodium, 50 ppm ammonium, 50 ppm potassium, 50 ppm magnesium, and 50 ppm calcium.

5.2.4.1 This standard stock can be prepared from 1000 ppm stock solutions using the following formula:

Combine 0.125 ml of the lithium standard and 1.25 ml of the sodium, ammonium, potassium, magnesium, and calcium standards in a 25 ml volumetric flask. Dilute to volume with 10% 2-propanol / 90% deionized water. The stock solution is stable for several weeks if kept refrigerated. Warm to room temperature before pipetting.

5.2.5 Prepare volumes of anion calibration standard solution for a three point calibration.

5.2.5.1 Standard 1: (0.05 ppm fluoride, 0.25 ppm acetate, 0.5 ppm formate, 0.2 ppm methanesulfonic acid (MSA), 0.5 ppm chloride, 0.2 ppm nitrite, 0.5 ppm bromide, 0.2 ppm nitrate, 2.0 ppm adipate, 1.0 ppm succinate, 0.2 ppm sulfate, and 0.2 ppm phosphate) Pipet 1 ml of the combination stock standard solution to a 100 ml volumetric flask. Add 20 microliters of 1000 ppm nitrite purchased stock solution and dilute to volume with 10% 2-propanol / 90% deionized water. Mix well. Prepare fresh daily. (Nitrite is not stable over long periods of time)

5.2.5.2 Standard 2: (0.1 ppm fluoride, 0.5 ppm acetate, 1.0 ppm formate, 0.4 ppm methanesulfonic acid (MSA), 1.0 ppm chloride, 0.4 ppm nitrite, 1.0 ppm bromide, 0.4 ppm nitrate, 4.0 ppm adipate, 2.0 ppm succinate, 0.4 ppm sulfate, and 0.4 ppm phosphate) Pipet 1 ml of the combination stock standard solution to a 50 ml volumetric flask. Add 40 microliters of 1000 ppm nitrite purchased stock solution and dilute to volume with 10% 2-propanol / 90% deionized water. Mix well. Prepare fresh daily. (Nitrite is not stable over long periods of time).

5.2.5.3 Standard 3: (0.2 ppm fluoride, 1.0 ppm acetate, 2.0 ppm formate, 0.8 ppm methanesulfonic acid (MSA), 2.0 ppm chloride, 0.8 ppm nitrite, 2.0 ppm bromide, 0.8 ppm nitrate, 8.0 ppm adipate, 4.0 ppm succinate, 0.8 ppm sulfate, and 0.8 ppm phosphate) Pipet 1 ml of the combination stock standard solution to a 25 ml volumetric flask. Add 80 microliters of 1000 ppm nitrite purchased stock solution and dilute to volume with 10% 2-propanol / 90% deionized water. Mix well. Prepare fresh daily. (Nitrite is not stable over long periods of time).

5.2.6 Prepare volumes of cation calibration standard solution for a three point calibration.

5.2.6.1 Standard 1: (0.05 ppm lithium, 0.5 ppm sodium, ammonium, potassium, magnesium, and calcium) Pipet 1 ml of the combination stock cation standard solution to a 100 ml volumetric flask and dilute to volume with 10% 2-propanol / 90% deionized water and mix well. Prepare fresh daily.

5.2.6.2 Standard 2: (0.1 ppm lithium, 1.0 ppm sodium, ammonium, potassium, magnesium, and calcium) Pipet 1 ml of the combination stock standard solution to a 50 ml volumetric flask and dilute to volume with 10% 2-propanol / 90% deionized water and mix well. Prepare fresh daily.

5.2.6.3 Standard 3: (0.2 ppm lithium, 2.0 ppm sodium, ammonium, potassium, magnesium, and calcium) Pipet 1 ml of the combination stock standard solution to a 25 ml volumetric flask and dilute to volume with 10% 2-propanol / 90% deionized water and mix well. Prepare fresh daily.

5.2.7 Run a minimum 3 point calibration per the chromatograph manufacturer's recommended methods for both anions and cations, verifying correct standard concentrations have been entered into the method.

5.2.8 Adjust the baselines and update the calibration as required to obtain a good calibration curve. The correlation factor (R^2) for the curves should be a minimum of 0.98, with higher values desirable. Any point on the calibration curve should not deviate from the expected value by more than ± 10%.

Note: The organic acids standards will typically not form a linear calibration and a quadratic curve may be required.

5.3 Analytical Procedure

5.3.1 The analysis of the extract solution should be done as soon as possible after extraction, but **shall** be no longer than four days from the extraction date.

5.3.2 Start the chromatograph per the manufacturers recommended method and allow it to come to a stable baseline.

5.3.3 Analyze sample solutions for anion and cation content, utilizing best analytical technique and laboratory practices.

5.4 Calculation of Results

5.4.1 Values from the chromatograms are typically reported in parts per million (ppm).

5.4.2 Surface Area Calculation Record the surface area of printed board (length x width x 2), e.g., a rectangular printed board with no cutouts. Alternatively, the surface area of the printed board can be determined from CAD software or other machine vision recognition system. Surface area should be known to three significant figures.

5.4.3 Results are to be expressed as micrograms (µg) of ion per square centimeter based on the extraction volume and the calculated sample surface area.

$$\mu g/cm^2 = \frac{(SC - BL) \times Vol}{Area}$$

where:

SC = ppm from IC (µg/mL)
BL = PPM from the bag blank
Vol = final volume (ml)
Area = surface area (cm^2)

Note: ''ppm'' value is actually specimen value minus blank value.

5.4.4 Report all ions quantified.

6 Notes

A repeatable and reproducible ionic cleanliness evaluation method requires some level of skill in accurately running an ion chromatography unit. The reader may find IPC-WP-008 to be of use.