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APPLICATION NOTE 1163

Determination of anions on the surface of printed circuit boards by IPC-TM-650 Method 2.3.28 using HPIC

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Keywords
Dionex IonPac AS11-HC-4µm column, Dionex ICS-5000⁺ HPIC system, Suppressed Conductivity

Goal

To develop an accurate method for determining specific anionic contamination on the surface of printed circuit boards (PCBs) as mentioned in the IPC method using a high-pressure ion chromatography (HPIC™) system with suppressed conductivity detection.

Introduction

The printed circuit board industry has long been interested in the ionic cleanliness of printed board surfaces and its correlation with corrosion, electrochemical migration, dendritic growth, and subsequent opens, leakage current, or shorting during testing or in the field. Initial methods for cleanliness evaluation included resistivity of solvent extract (ROSE), which measured the conductivity of a solution after flowing it over a surface of interest. A major disadvantage of this technique was its inability to detect the specific ionic species generating the measured conductivity.

Ion chromatography (IC) has become an important technique for the evaluation of ionic cleanliness. This technique, which detects individual ions, allows quicker troubleshooting of contamination sources and better predictions about the detrimental effects of each ionic species. It is able to measure concentrations of major anions, such as chloride and bromide, as well as major cations such as sodium, ammonium, and potassium at low parts-per-million (ppm) and even parts-per-billion (ppb) concentrations. Concentrations of weak organic acids (WOAs) can also be measured by IC.

This application note describes an IC method using a Thermo Scientific™ Dionex™ IonPac™ AS11-HC-4µm column and a Thermo Scientific™ Dionex™ ICS-5000⁺ HPIC system to determine extractable anionic contaminants on the

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surface of PCBs, including inorganic anions and WOAs following the extraction procedure described in IPC-TM-650 Method 2.3.28.[1]

The 15 anions mentioned in the IPC Method are bromide, chloride, fluoride, nitrate, nitrite, phosphate, sulfate, acetate, adipate, formate, glutamate, malate, methanesulfonate, succinate, and phthalate. Our method addressed all anions except for glutamate, which contains a α-amino group, a α-carboxylic acid group, and a side chain carboxylic acid, which may mean that it is removed during the suppression process.

Equipment

- Dionex ICS-5000+ HPIC system, including:
  - Dionex ICS-5000+ DP Pump module
  - Dionex ICS-5000+ EG Eluent Generator module with high-pressure degasser module
  - Dionex ICS-5000+ DC Detector/Chromatography module
- Thermo Scientific™ Dionex™ AS-AP Autosampler with Sample Syringe, 250 µL (P/N 074306) and Buffer line, 1.2 mL (P/N 074989)
- Thermo Scientific™ Dionex™ EGC 500 KOH Eluent Generator Cartridge (P/N 075778)
- Thermo Scientific™ Dionex™ CR-ATC 500 Continuously Regenerated Anion Trap Column (P/N 075550)
- Thermo Scientific™ Dionex™ AERS 500e Suppressor (Dionex AERS 500e (2 mm), P/N SP6953)
- 4 L water bottle (P/N 039164)
- Thermo Scientific™ Chromeleon™ Chromatography Data System software version 7.2

Reagents and standards

Reagents

Deionized (DI) water, Type I reagent grade, 18 MΩ-cm resistivity or better filtered through a 0.2 µm filter immediately before use, 2-Propanol (Certified ACS), Fisher Chemical (Fisher Scientific P/N A416-4).

Standards

- Sodium Bromide, (Granular/Certified ACS), Fisher Chemical (Fisher Scientific P/N S255-500)
- Sodium Chloride, (Crystalline/Certified ACS), Fisher Chemical (Fisher Scientific P/N S271-500)
- Sodium Fluoride, (Powder/Certified ACS), Fisher Chemical (Fisher Scientific P/N S299-500)
- Sodium Nitrate, (Crystalline/Certified ACS), Fisher Chemical (Fisher Scientific P/N S343-500)
- Sodium Nitrite, (Crystalline/Certified ACS), Fisher Chemical (Fisher Scientific P/N S347-500)
- Sodium Phosphate Monobasic Anhydrous (USP), Fisher Chemical (Fisher Scientific P/N S397-500)
- Sodium Sulfate Anhydrous, (Granular/Certified ACS), Fisher Chemical (Fisher Scientific P/N S421-500)
- Acetic Acid, Glacial (Certified ACS Plus), Fisher BioReagents (Fisher Scientific P/N BP 2401-500)
- Adipic Acid, (Powder/Certified), Fisher Chemical (Fisher Scientific P/N A44-500)
- Formic Acid, 99%, for analysis, ACROS Organics™ (Fisher Scientific P/N AC 270480250)
- L-(+)-Glutamic Acid, (Powder/Certified), Fisher Chemical (Fisher Scientific P/N A125-100)
- L(-)-Malic Acid, 99% (Fisher Scientific P/N AC15059)
- Methanesulfonic acid, 99%, extra pure, ACROS Organics™ (Fisher Scientific P/N AC125610050)
- Succinic Acid (Crystalline/Certified), Fisher Chemical (Fisher Scientific P/N A294-500)
- Potassium hydrogen phthalate, 99+%, Acros Organics™ (Fisher Scientific P/N AC417955000)
- Methanol (Optima™ LC/MS), Fisher Chemical (Fisher Scientific P/N A456-1)

Consumables

- Ampac™ 500 Series SealPAK Heavy Duty Pouches, 4.5 mils thick, 8 x 6.5 in. (Fisher Scientific P/N 01-812-25E) and 16 x 12 in. (Fisher Scientific P/N 01-812-25G)
- Vial Kit, 10 mL Polystyrene with Caps and Blue Septa (P/N 074228)
- Thermo Scientific™ Nalgene™ Syringe Filters, PES, 0.2 µm (Fisher Scientific P/N 09-740-61A)
- AirTite All-Plastic Norm-Ject™ Syringes, 5 mL, Sterile (Fisher Scientific P/N 14-817-28)
- Thermo Scientific Nalgene 1000 mL, 0.2 µm Nylon Filter Units (P/N 09-740-46)
- Fisherbrand™ Powder-Free Nitrile Exam Gloves (P/N 19-130-1597)

Samples*

- PCB Assembly A
- PCB Assembly B
- PCB Assembly C

* Samples came from scrap at Thermo Fisher Scientific in California.

Preparation of Solutions and Reagents

Deionized (DI) water was used for eluent and standard preparation and for diluting samples. Individual stock standard solutions of 1000 mg/L were prepared gravimetrically from the reagents and DI water. A mixed standard solution was prepared by diluting the individual stock standard solutions into a 100 mL volumetric flask with DI water. Calibration standards were prepared similarly by diluting the stock standards in DI water. The 20 compounds listed in Table 1 were used to prepare 100 mL of 1000 mg/L stock solutions.

Table 1. Amounts of compounds used to prepare 100 mL of 1000 mg/L stock solutions.

Anion	Compound	Mass (mg)
Bromide	Sodium Bromide	128.77
Chloride	Sodium Chloride	164.85
Fluoride	Sodium Fluoride	221.01
Nitrate	Sodium Nitrate	137.08
Nitrite	Sodium Nitrite	149.96
Phosphate	Sodium Phosphate, Monobasic	126.33
Sulfate	Sodium Sulfate	147.87
Acetate	Acetic Acid	100.00
Adipate	Adipic Acid	100.00
Formate	Formic Acid	100.00
Glutamate	Glutamic Acid	100.00
Malate	Malic Acid, Disodium Salt	132.78
Methane-sulfonate	Methanesulfonic Acid	100.00
Succinate	Succinic Acid	100.00
Phthalate	Potassium Hydrogen Phthalate	124.44

Methanol was degassed by ultrasonic agitation. Degassed methanol was added to a second 1 L eluent bottle on Channel B under inert atmosphere and introduced into the eluent through the proportioning valve.

Conditions		
Columns:	Dionex IonPac AG11-HC-4-µm Guard, 2 × 50 mm (P/N 078036)	
	Dionex IonPac AS11-HC-4-µm Analytical, 2 × 250 mm (P/N 078035)	
Eluent Source:	Dionex EGC 500 KOH Eluent Generator Cartridge with CR-ATC 500	
Eluent A:	DI Water	
Eluent B:	Methanol (CH_3OH)	
Gradient:		
Time (min)	KOH (mM)	
-5.00	1	
0.00	1	
17.00	1	
24.00	15	
35.30	15	
54.60	60	
54.61	1	
55.00	1	
Pump_1:	Multi-Step Gradient	
Time (min)	B (%)	Curve
-5.00	0	5
0.00	0	5
22.00	0	5
24.00	10	5
39.30	12	5
51.00	10	5
54.60	0	5
55.00	0	5
Pump_1 Flow Rate:	0.38 mL/min	
Pump_2:	Isocratic (delivers external water for the Dionex AERS 500e suppressor)	
Pump_2 Flow Rate:	0.76 mL/min	
Injection Vol.:	5 µL	
Temperature:	40 °C (column compartment), 25 °C (detector compartment)	
System Backpressure:	~4000 psi (1 mM KOH/0% CH_3OH), ~4900 psi (22 mM KOH/12% CH_3OH)	
Detection:	Suppressed Conductivity, Dionex AERS 500e Electrolytically Regenerated Suppressor (2 mm), AutoSuppression, 57 mA, external water mode	
Background Conductance:	~ 0.5 µS	
Run Time:	60 min	

Sample preparation

The ion extraction process is based on that described in IPC-TM-650 method 2.3.28. The steps are as follows:

1. Select a low-ion extraction bag sized to fit the board with approximately 2.5 cm [1.0 in] excess on each side to minimize the required extract solution, with several inches at the top to allow for air expansion when the bag is heated.
2. Place boards and panels into clean Ampac 500 series Sealpak heavy duty pouches using clean nitrile gloves.
3. Prepare a 75/25 (+/- 5%) v/v IPA/H_20 solution for the extraction.
4. Add a known volume of the extraction solution to the extraction bag, covering the sample (approximately 0.5 mL/cm^2 of surface area).
5. Add the same volume of extraction solution to an empty bag of the same lot for use as a blank.
6. Heat seal all sample and blank extraction bags and place in an 80 °C [176 °F] water bath for 1h and no longer than 65 min.
7. Allow the solution in the bag to cool to ambient temperature before opening.
8. Record the surface area of PCB (length x width x 2) or PCA. As a general rule for assemblies, the surface area is estimated as: (length x width x 2) + 10%. Great caution should be taken in interpretation and comparison of these results as assembly surface areas often deviate by more than 10% of their unpopulated state (i.e., if the board was flat with no components).
9. Gently mix the contents. Transfer solution to pass through a Nalgene syringe filter (0.2 µm) and store at 4 °C prior to analysis.

Recovery study

Three anions were selected for the recovery study – acetate, adipate, and sulfate – which are distributed in the early, middle, and late retention regions of the separation, respectively. To ensure that our measurement was accurate, the samples were spiked with appropriate known amounts of stock solution.

Dionex ICS-5000+ HPIC DP system preparation and configuration

The Dionex AS-AP Autosampler was installed and configured in Push Mode. Following the instructions in the Dionex AS-AP Autosampler Operator's Manual (Document No. 065361) the sample transfer line was calibrated to ensure accurate and precise sample injections.

The Dionex EGC 500 KOH cartridge, Thermo Scientific Dionex CR-ATC 500 Continuously Regenerating Anion Trap Column, and Dionex AERS 500e suppressor were installed and hydrated according to the product manual instructions.[2–4]

Note: When methanol is added to the eluent stream, the suppressor must be operated in the External Water mode to prevent contamination from methanol hydrolysis and potential damage to the suppressor. External water for Dionex AERS 500e suppressor regeneration can be delivered via the second pump of the DP module as in this study or a pressurized water reservoir.[5]

The Dionex IonPac AG11-HC-4µm Guard (2 × 50 mm) and the Dionex IonPac AS11-HC-4µm Analytical (2 × 250 mm) columns were installed in the lower compartment of the DC detector. After connecting the inlet of the column, 30 mM KOH was pumped through the column with the outlet directed to waste for at least 30 min before connecting the column outlet to the suppressor using 0.005 in. i.d. PEEK tubing. The lengths of connective tubing were kept to a minimum. Performance of the Dionex IonPac AS11-HC-4µm column was evaluated by injecting the quality assurance report (QAR) standard mix. The column is equilibrated when at least three consecutive injections of the standard produce the same RTs for all analytes.

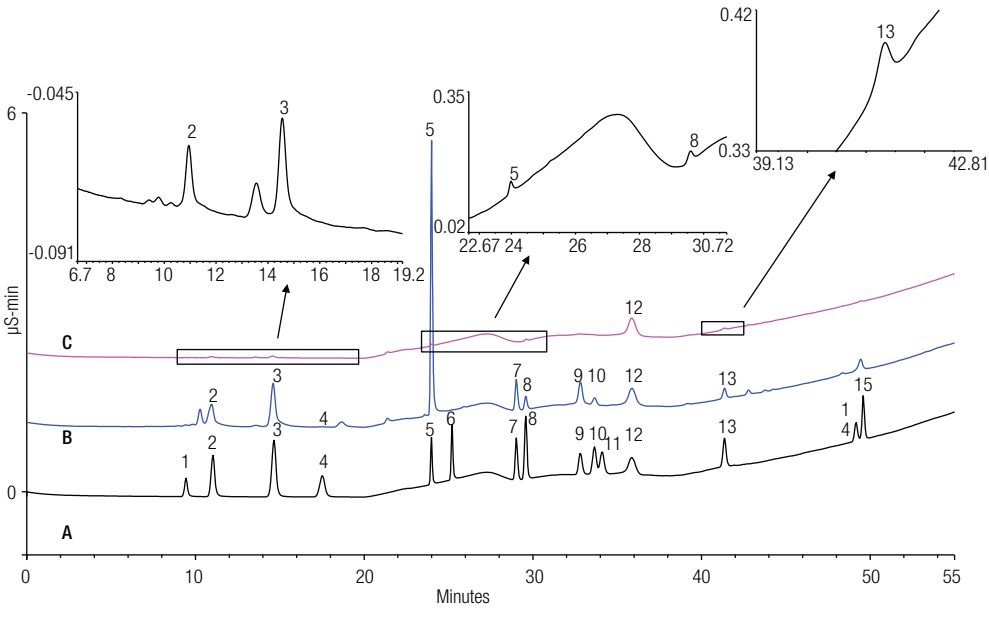
It was confirmed that the resulting chromatogram resembled the chromatogram shown in the QAR that comes with the column. Note that the chromatogram shown in the QAR is generated without the guard column; therefore, analyte RTs should be longer than those shown in the QAR.

Results and discussion

Separation

The Dionex IonPac AS11-HC-4µm column is a high resolution, high-capacity anion exchange column, providing separations for the best peak identification of a large number of inorganic anions and organic acid anions from a single sample injection. The column was operated in gradient mode using a hydroxide eluent. Certain organic solvents can be added to the hydroxide eluent to modify the ion exchange process, and thereby column selectivity, or to improve sample solubility. Under aqueous eluent conditions, succinate and malate co-elute. In order to improve the separation of malate and succinate, methanol can be added to the eluent. Note that when adding methanol to the eluent stream, the suppressor must be operated in the External Water mode. Adding solvent to the aqueous eluent can reduce the peak response by conductivity by up to half due to increased eluent viscosity, decreased ionization of organic acids, and in some cases, lower peak efficiencies. Therefore, solvent was used in the eluent only when needed for improved analyte resolution.

In this study, a hydroxide-methanol gradient was used to separate 14 anions on the Dionex IonPac AS11-HC-4µm column set. (Figure 1, Chromatogram A) The method started with a low eluent concentration (1 mM KOH) to separate the weakly retained anions, such as fluoride and acetate. Due to unknown peaks interfering with methane sulfonate in some PCB samples, the eluent concentration was maintained at 1 mM KOH to resolve those peaks at 17 min. The eluent concentration was then gradually increased to elute more strongly retained anions. Methanol was added to resolve succinate and malate for accurate quantification because they are typically monitored in PCB samples. Methanol introduction started at 22 min, and the percentage of methanol was ramped to 10% at 24 min, increased to 12% at 39.3 min, and returned to 10% at 51 min. The eluent condition was restored to the initial condition at 54.6 min to re-equilibrate the column prior to the next injection. To reduce the high backpressure when running eluents with methanol, the column was operated at 40 °C.



Peaks	Standard	PCB Assembly A mg/L	PCB Blank A
1. Fluoride	0.250	—	—
2. Acetate	2.50	1.69	0.111
3. Formate	2.50	2.41	0.135
4. Methanesulfonate	2.50	0.251	—
5. Chlorlde	0.750	3.47	0.133
6. Nitrite	1.25	—	—
7. Bromide	2.50	1.93	—
8. Nitrate	2.50	0.633	0.134
9. Adlpate	2.50	2.87	—
10. Succinate	2.50	0.474	—
11. Malate	2.50	—	—
12. Carbonate	n.a.	n.a.	n.a.
13. Sulfate	1.25	0.471	1.16
14. Phthalate	2.50	—	—
15. Phosphate	3.75	—	—

"-"= < LOQ
n.a. = not applicable

Figure 1. Separation of 14 organic and inorganic anions on the Dionex IonPac AS11-HC-4µm column in (A) a mix of 14 anion standards (complete conditions shown in the Conditions section), (B) PCB assembly A, and (C) PCB blank A with enlarged sections of the chromatogram so that the peaks can be observed.

Calibration, limit of detection, and limit of quantitation

The calibration curves were constructed for 14 anions that are recommended for evaluation in IPC-TM-650 Method 2.3.28. Calibration curves with seven concentration levels ranging from 0.1 mg/L to 10 mg/L were constructed for acetate, formate, chloride, nitrate, bromide, and nitrate, from 0.05 mg/L to 5 mg/L for fluoride, from 0.2 mg/L to 20 mg/L for methane sulfonate, from 0.4 mg/L to 50 mg/L for adipate, from 0.5 mg/L to 50 mg/L for succinate, from 0.5 mg/L to 10 mg/L for malate and phthalate, from 0.1 mg/L to 5 mg/L for sulfate, and from 0.2 mg/L to 4 mg/L for phosphate (Table 2).

Due to incomplete dissociation of these weak carboxylic acids at high concentrations, the calibration curves show deviation from linearity in the selected calibration ranges.[6] Therefore, the calibration plots of peak area versus concentration were fit using quadratic regression functions with coefficients of determination (r^2) >0.999. Determination of the signal-to-noise ratio was performed by comparing measured signals from samples with known low concentrations of analyte with those of blank samples and by establishing the minimum concentration at which the analyte can be reliably detected. A signal-to-noise ratio of 3:1 is generally considered acceptable for estimating the limit of detection (LOD), and a signal-to-noise ratio 10:1 for limit of quantification (LOQ). The LODs and LOQs were then calculated from the average peak height of five injections of 0.2 mg/L adipate, succinate, malate, phthalate, and phosphate, and 0.1 mg/L for fluoride, acetate, formate, methanesulfonate, chloride, nitrite, bromide, nitrate, and sulfate. The results of the calibration, LOD, and LOQ are summarized in Table 2.

Table 2. Method calibration, LOD, and LOQ.

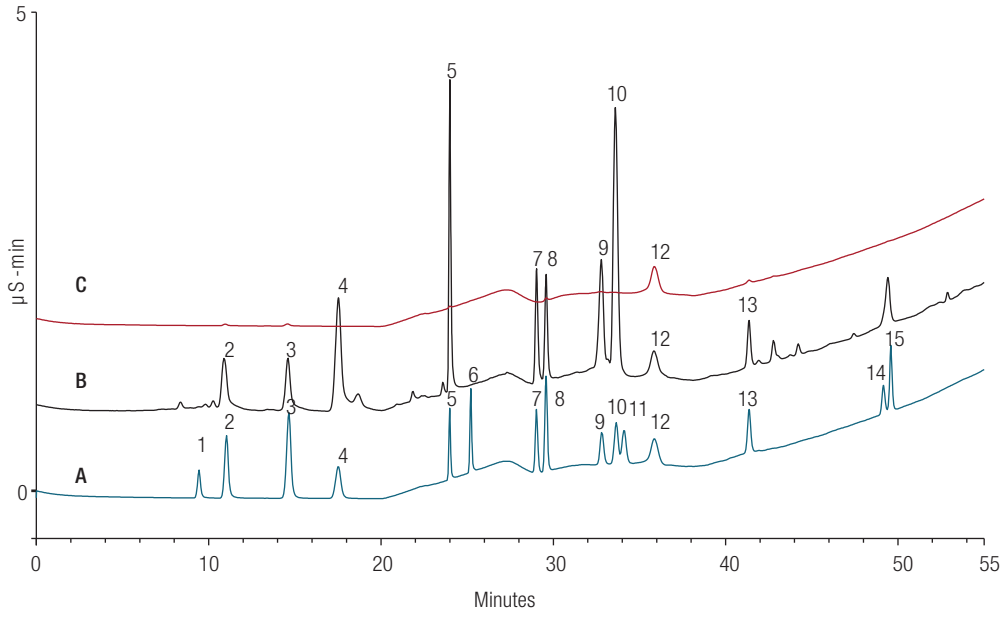
Compound	Range (mg/L)	Coefficient of Determination (r^2)	LOD[b] (mg/L)	LOQ[c] (mg/L)
Fluoride	0.05–5	0.9999	< 0.01	< 0.04
Acetate	0.1–10	1.000[a]		
Formate	0.1–10	0.9999[a]		
Methane-sulfonate	0.2–20	0.9996		
Chloride	0.1–10	0.9997		
Nitrite	0.1–10	0.9992		
Bromide	0.1–10	0.9999[a]	0.033	0.109
Nitrate	0.1–10	0.9999[a]	0.022	0.073
Adipate	0.4–50	0.9999[a]	0.115	0.383
Succinate	0.5–50	0.9999[a]	0.096	0.319
Malate	0.5–10	0.9999[a]	0.143	0.478
Sulfate	0.1–5	0.9991	0.027	0.091
Phthalate	0.5–10	0.9999[a]	0.089	0.295
Phosphate	0.2–4	0.9996	0.059	0.197

[a]Quadratic fit
[b]LOD=3×S/N
[c]LOQ=10×S/N

Sample analysis

In Figures 1, 2, and 3, chromatograms B and C show the analysis of PCB assembly samples and blanks. Acetate, formate, chloride, nitrate, and sulfate are the main anions found in blank ionic extractions. There are slight differences in anionic composition and content of PCB samples. The various organic acids were identified by comparing their retention times with those of standards. Chloride and bromide are the most common inorganic contaminants in PCB assembly failures, while WOA levels vary greatly, depending on the delivery method (e.g., foam or spray) and the preheat dynamics. In our study, no nitrite, phthalate, or phosphate were detected in PCB assembly samples.

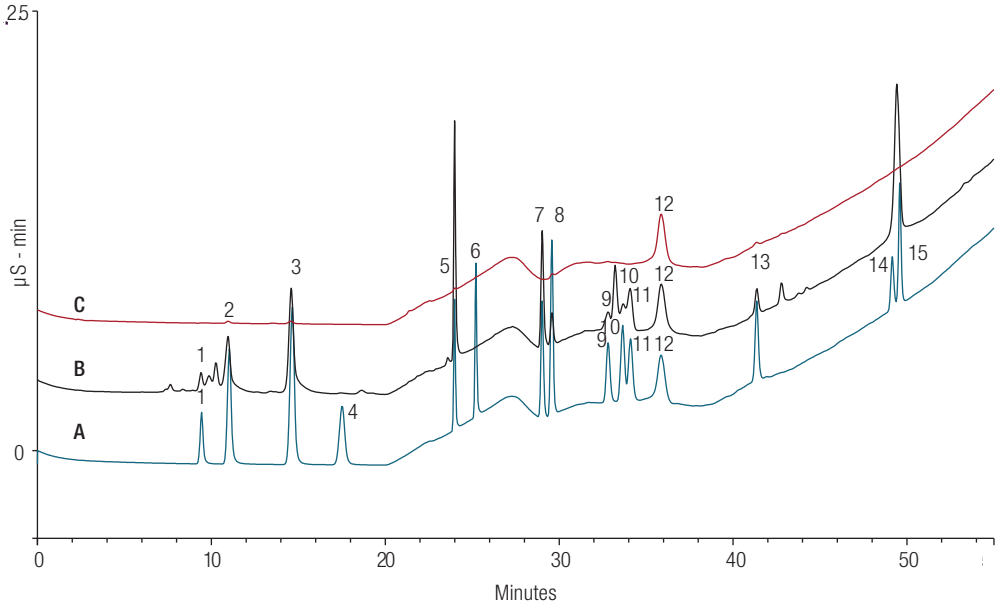
The levels of all the anions listed in Figures 1, 2, and 3 were determined from their respective calibration curves.



Peaks	Standard	PCB Assembly A mg/L	PCB Blank A
1. Fluoride	0.250	—	—
2. Acetate	2.50	3.08	0.0993
3. Formate	2.50	1.99	0.112
4. Methanesulfonate	2.50	8.42	—
5. Chloride	0.750	2.76	0.123
6. Nitrite	1.25	—	—
7. Bromide	2.50	4.43	—
8. Nitrate	2.50	2.83	0.147
9. Adlpate	2.50	9.64	—
10. Succinate	2.50	19.2	—
11. Malate	2.50	—	—
12. Carbonate	n.a.	n.a.	n.a.
13. Sulfate	1.25	1.27	0.118
14. Phthalate	2.50	—	—
15. Phosphate	3.75	—	—

"-"= < LOQ
n.a. = not applicable

Figure 2. Separation of PCB assembly B and PCB blank B on the Dionex IonPac AS11-HC-4 µm column in (A) a mix of 14 anion standards (complete conditions shown as in the Conditions section), (B) PCB assembly B, and (C) PCB blank B.



Peaks	Standard	PCB Assembly A mg/L	PCB Blank A
1. Fluoride	0.250	0.0903	—
2. Acetate	2.50	1.45	0.0999
3. Formate	2.50	1.92	0.103
4. Methanesulfonate	2.50	0.252	—
5. Chlorlde	0.750	1.17	0.118
6. Nitrite	1.25	—	—
7. Bromide	2.50	2.59	—
8. Nitrate	2.50	0.545	0.113
9. Adlpate	2.50	0.679	—
10. Succinate	2.50	—	—
11. Malate	2.50	1.19	—
12. Carbonate	n.a.	n.a.	n.a.
13. Sulfate	1.25	0.440	—
14. Phthalate	2.50	—	—
15. Phosphate	3.75	—	—

"-"= < LOQ
n.a. = not applicable

Figure 3. Separation of PCB assembly C and PCB blank C on the Dionex IonPac AS11-HC-4 µm column in (A) a mix of 14 anion standards (complete conditions shown in the Conditions section), (B) PCB assembly C, and (C) PCB blank C.

Precision

The precision of an analytical procedure is usually expressed as the relative standard deviation (RSD) of a series of measurements. For our method, the peak area and retention time precisions were determined for seven replicate injections of a standard mixture containing 2.5 mg/L acetate, 2.5 mg/L adipate, and 1.25 mg/L sulfate. The retention time RSDs and the peak area RSDs of the three representative analytes are within 0.1% and 3% respectively (Table 3), indicating good method precision at these analyte concentrations.

Table 3. Method precision.

Analyte	RT (min)	RT RSD	Area (nC*min)	Peak Area RSD
Acetate	11.04	0.08	0.18	0.33
Adipate	32.82	0.01	0.09	2.16
Sulfate	41.37	0.01	0.10	1.14

Accuracy

The accuracy of our method was verified by determining recoveries of acetate, adipate, and sulfate in spiked PCB samples (Table 4). Samples were spiked with standard solutions at a series of percentages (20, 50, 100, or 150) of the amount measured. Recoveries were calculated from the difference in response between the spiked and unspiked samples. The average recovery for three anions ranged from 87 to 114%, indicating that this method can accurately determine anions in PCB samples.

Table 4. Recovery of acetate, adipate, and sulfate.

PCB assembly A			
	Acetate (mg/L)	Adipate (mg/L)	Sulfate (mg/L)
*Endogenous	2.06	2.87	0.431
Spiked			
20% addition	0.36	0.500	0.0800
50% addition	0.72	1.00	0.160
100% addition	1.44	2.00	0.320
150% addition	2.16	3.00	0.480
Measured			
20% addition	0.393	0.465	0.0785
50% addition	0.660	1.07	0.146
100% addition	1.25	2.12	0.293
150% addition	1.88	3.02	0.498
Recovery (%)			
20% addition	109	93	98
50% addition	92	107	91
100% addition	87	106	91
150% addition	87	101	104

Table 4. Recovery of acetate, adipate, and sulfate.

PCB assembly B			
	Acetate (mg/L)	Adipate (mg/L)	Sulfate (mg/L)
*Endogenous	3.05	10.5	1.24
Spiked			
20% addition	0.520	1.85	0.240
50% addition	1.04	3.70	0.480
100% addition	2.08	7.40	0.960
150% addition	3.12	11.1	1.44
Measured			
20% addition	0.559	1.95	0.223
50% addition	1.13	3.62	0.499
100% addition	2.29	7.19	1.00
150% addition	3.26	10.4	1.47
Recovery (%)			
20% addition	107	105	93
50% addition	108	98	104
100% addition	110	97	104
150% addition	104	94	102
PCB assembly C			
	Acetate (mg/L)	Adipate (mg/L)	Sulfate (mg/L)
*Endogenous	1.86	0.659	0.403
Spiked			
20% addition	0.290	0.165	0.0800
50% addition	0.580	0.330	0.160
100% addition	1.16	0.660	0.320
150% addition	1.74	0.990	0.480
Measured			
20% addition	0.326	0.167	0.0910
50% addition	0.582	0.344	0.179
100% addition	1.09	0.682	0.280
150% addition	1.62	1.02	0.528
Recovery (%)			
20% addition	113	101	114
50% addition	100	104	112
100% addition	94	103	88
150% addition	93	103	110

Contamination calculation

The following calculations are used to determine the weight of the anionic contaminants per unit area.[1] Results are to be expressed as µg of ion per square centimeter based on the extraction volume and the calculated sample surface area. The level in the blank is subtracted from that found in the sample:

$$\mu g/cm^2 = \frac{[C_s\ (\mu g/mL) - C_b\ (\mu g/mL)] \times [extract\ volume\ (mL)]}{[surface\ area\ (cm^2)]}$$

Table 5. Contamination levels of 14 anions in three PCB samples.

Analyte	PCB assembly A	PCB assembly B	PCB assembly C
	µg/cm²		
Fluoride	–	–	0.0204
Acetate	0.362	0.680	0.305
Formate	0.522	0.427	0.411
Methanesulfonate	0.058	1.92	0.0568
Chloride	0.766	0.601	0.236
Nitrite	–	–	–
Bromide	0.443	1.01	0.583
Nitrate	0.114	0.612	0.0974
Adipate	0.658	2.20	0.153
Succinate	0.109	4.39	
Malate	–	–	–
Sulfate	0.081	0.263	0.0993
Phthalate	–	–	–
Phosphate	–	–	–

Where C_s is the concentration in the sample, C_b is the concentration in the blank, and C_s-C_b is the blank-corrected concentration for the sample. The contamination levels were calculated in Table 5.

Conclusion

This study developed an accurate method to determine 14 anions that can be present on the surface of a PCB. The method uses a Dionex IonPac AS11-HC-4µm column combined with suppressed conductivity detection on a HPIC system for optimal separation and quantification of inorganic anions and organic acids in PCB extraction samples. The specificity and sensitivity of this method allow us to measure the concentrations of bromide, chloride, fluoride, nitrate, nitrite, phosphate, sulfate, acetate, adipate, formate, glutamate, malate, methane sulfonate, succinate, and phthalate. The recovery study demonstrated good method accuracy. A gradient pump allowed the addition of a methanol gradient to the eluent-generator-produced hydroxide gradient, resulting in the separation of succinate and malate, which coelute under aqueous eluent conditions.

REFERENCES

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